

FORM PTO-1390
(REV. 11-2000)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

02365

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

09/936756

INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/EP00/02258

15 March 2000 (15.03.00)

15 March 1999 (15.03.99)

TITLE OF INVENTION METHOD FOR PRODUCING PHYSICALLY FOAMED INJECTION MOULDED PARTS

APPLICANT(S) FOR DO/EO/US ULRICH, STIELER

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☐ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☒ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☒ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:
English translation of the application as amended.
International Preliminary Examination Report including amendments (in German)

U.S. APPLICATION NO. (if known, see 37 CFR 1.53) 09/936756	INTERNATIONAL APPLICATION NO PCT/EP00/02258	ATTORNEY'S DOCKET NUMBER 02365
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21. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a) (2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO **\$1000.00**

International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO **\$860.00**

International preliminary examination fee (37 CFR 1.482) not paid to USPTO
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO **\$710.00**

International preliminary examination fee (37 CFR 1.482) paid to USPTO
but all claims did not satisfy provisions of PCT Article 33(1)-(4) **\$690.00**

International preliminary examination fee (37 CFR 1.482) paid to USPTO
and all claims satisfied provisions of PCT Article 33(1)-(4) **\$100.00**

ENTER APPROPRIATE BASIC FEE AMOUNT =

CALCULATIONS PTO USE ONLY

\$ 860.00

Surcharge of **\$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(e)).

\$ 0.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	\$
Total claims	24 -20 =	4	x \$18.00	\$ 72.00	
Independent claims	3 -3 =	0	x \$80.00	\$ 0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$270.00	\$ 0.00	
TOTAL OF ABOVE CALCULATIONS =				\$ 932.00	

☒ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above
are reduced by 1/2. +

\$ 466.00

SUBTOTAL =

\$ 466.00

Processing fee of **\$130.00** for furnishing the English translation later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$ 0.00

TOTAL NATIONAL FEE =

\$ 466.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **\$40.00** per property +

\$ 0.00

TOTAL FEES ENCLOSED =

\$ 466.00

Amount to be
refunded: \$

charged: \$

a. ☒ A check in the amount of \$ 466.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees.
A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any
overpayment to Deposit Account No. 19-0120. A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card
information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR
1.137 (a) or (b)) must be filed and granted to restore the application to pending status.**

SEND ALL CORRESPONDENCE TO:

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SIGNATURE

Michele J. Young
NAME

43,299

REGISTRATION NUMBER

Express Mail Label No. EL 775785023 US

ATTORNEY DOCKET NO. 02365
IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicant : Ulrich STIELER

Serial No. : Unknown

Filed : Herewith

Title : METHOD FOR PRODUCING PHYSICALLY FOAMED INJECTION
MOULDED PARTS

Assistant Commissioner for Patents
Washington DC 20231

PRELIMINARY AMENDMENT

Sir/Madam:

Prior to examination of the above-identified application, entry of this preliminary amendment is respectfully requested. Please amend the above-identified application as follows:

In the Specification:

Please amend the specification as follows:

Please replace the specification with the substitute specification, excluding claims, submitted herewith under 37 C.F.R. 1.125(b).

In the Claims:

Please rewrite the claims as follows:

1. (Amended Once) A process for the production of physically foamed injection molded articles, wherein in a first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion

(propellant injection phase), and possibly in a third stage a propellant-free further melt portion is charged into the cavity, the production of the injection molded articles occurring in the cavity,

wherein metering of the physical propellant in the second stage occurs in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity.

2. (Amended Once) The process of Claim 1, wherein the propellant is a compressible fluid.
3. (Amended Once) The process of Claim 1 further comprising the step of maintaining the propellant under pressure in the intermediate cycle times before and after the propellant injection phase.
4. (Amended Once) The process of Claim 3, further comprising maintaining the propellant at a pressure of at least p_{crit} at a given temperature during the intermediate cycle times.
5. (Amended Once) The process of Claim 1, further comprising the step of controlling the pressure exerted on the propellant via a pressure control valve.
6. (Amended Once) The process of Claim 5, wherein the pressure control valve is a multi-way valve.
7. (Amended Once) The process of Claim 6, wherein the multi-way valve is a 3/3-way proportional valve or a 2/3-way proportional valve.

8. (Amended Once) The process of claim 1 further comprising the step of controlling the pressure of the critical propellants via at least one pressure relief valve connected downstream of the pressure control valve.
9. (Amended Once) The process of Claim 8, wherein at least one of the pressure relief valves has a holding pressure equal to or higher than the pressure at which a critical propellant is held in the intermediate cycle times.
10. (Amended Once) The process according to Claim 1 further comprising the step of regulating the pressure preset by the pressure control valve via one or more pressure relief valves to the injection pressure at which the propellant is added to the melt via an injection point.
11. (Amended Once) The process of claim 1, wherein the injection point is configured as a throttle means.
12. (Amended Once) The process of Claim 11, wherein the injection point is in the form of a defined gap in an injector or of an injector with a sinter metal.
13. (Amended Once) The process of Claim 11, wherein the injection point is configured as a controlled closure mechanism.
14. (Amended Once) The process of Claim 1 further comprising the step of using water as the propellant.
15. (Amended Once) The process of Claim 1 further comprising the step of using a gas or gas mixture as the propellant.

16. (Amended Once) The process of Claim 15, further comprising the step of using carbon dioxide as the propellant.
17. (Amended Once) The process of Claim 16, wherein the carbon dioxide is held in the intermediate cycle times at a pressure of at least 60 bar.
18. (Amended Once) The process of Claim 1 further comprising the step of elevating the pressure of the propellant during the propellant injection phase to a pressure of over 60 bar using the pressure control valve.
19. (Amended Once) The process of Claim 1 further comprising the step of generating a counterpressure in the cavity.
20. (Amended Once) The process of Claim 1, wherein the physically foamed injection molded article is selected from the group consisting of a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a fender, a float and a closing means for bottle-like containers.
21. (Amended Once) A device for the metered addition of physical propellants to a foamable melt, comprising:
- a storage means, in which the propellant is stored under pressure,
 - a pressure control valve for regulating the propellant pressure, and
 - an injection point, which is configured as a throttle means, at which the propellant under pressure is fed to the melt,
- wherein a controlled closure mechanism is provided at the injection point.

22. (Amended Once) The device of Claim 21, further comprising at least one pressure relief valve.

Please add the following claims:

--23. (New) The process of claim 1, further comprising the step of:
maintaining the propellant in a compressed state in the intermediate cycle times before and after the propellant injection phase.

24. (New) A device for the metered addition of physical propellants to a foamable melt, comprising:

a storage means, in which the propellant is stored under pressure,
a pressure control valve for regulating the propellant pressure, and
an injection point, which is configured as a throttle means, at which the propellant under pressure is fed to the melt,

wherein at least one pressure relief valve is provided at the injection point.--

REMARKS

By way of this Preliminary Amendment, the English translation of the Specification has been amended to conform to U.S. Practice. A Substitute Specification excluding claims under 37 C.F.R. 1.125(b) is submitted herewith accompanied by a marked-up copy of the specification showing the matter being added to and the matter being deleted from the specification of record. The Substitute Specification does not include new matter.

In addition, by the present amendment, claims 1-22 have been amended to conform to U.S. Practice. These amendments are not considered to narrow the scope of the claims. Claims 23 and 24 have been added, and are directed to alternative elements of original claims 3 and 22.

The Applicant respectfully submit that no new matter has been added by this Preliminary Amendment, and respectfully requests entry of this preliminary amendment.

CONCLUSION

In view of the foregoing amendments and remarks, the Applicant respectfully submits that the pending claims in the above-identified application are in condition for allowance, and a notice to that effect is earnestly solicited.

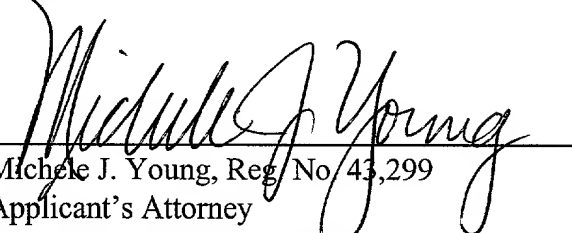
If the present application is found by the Examiner not to be in condition for allowance, then the Applicant hereby requests a telephone or personal interview to facilitate the resolution of any remaining matters. Applicant's attorney may be contact by telephone at the number indicated below to schedule such an interview.

Applicant: STIELER, Ulrich
U.S. National Phase of PCT /EP 00/02258

The Patent and Trademark Office is authorized to charge any additional fees incurred as a result of the filing hereof or credit any overpayment to our Deposit Account No. 19-0120.

Respectfully submitted,
STIELER, Ulrich, Applicant

By:


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Dated: September 17, 2001

Version with marking to show changes made:

1. (Amended Once) A [P]process for the production of physically foamed injection [moulded] molded articles, wherein in a first stage a propellant-free first melt portion [(6)] is fed into a cavity [(1)] (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), and possibly in a third stage a propellant-free further melt portion is charged into the cavity [(1)], the production of the injection [moulded] molded articles occurring in the cavity,

[characterised in that] wherein metering of the physical propellant in the second stage occurs in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity [(1)].

2. (Amended Once) The [P]process [according to] of Claim 1, [characterised in that] wherein the propellant is a compressible fluid.

3. (Amended Once) The [P]process [according to] of Claim 1 [or 2, characterised in that] further comprising the step of maintaining the propellant [is kept] under pressure in the intermediate cycle times before and after the propellant injection phase[, or is present in a compressed state].

4. (Amended Once) The [P]process [according to] of Claim 3, [characterised in that in] further comprising maintaining the propellant at a pressure of at least p (crit) at a given temperature during the intermediate cycle times [the propellant is held a pressure of at least p (crit) of the propellant at the given temperature].

5. (Amended Once) The [P]process [according to one of the preceding claims] of Claim 1, [characterised in that] further comprising the step of controlling the pressure exerted on the propellant [is controlled] via a pressure control valve [(10)].

6. (Amended Once) The [P]process [according to] of Claim 5, [characterised in that] wherein the pressure control valve [(10)] is a multi-way valve.

7. (Amended Once) The [P]process [according to] of Claim 6, [characterised in that] wherein the multi-way valve is a 3/3-way proportional valve or a 2/3-way proportional valve [is used as multi-way valve].

8. (Amended Once) The [P]process [according to one of the preceding claims, characterised in that] of claim 1 further comprising the step of controlling the pressure [control in the case] of the critical propellants [additionally occurs] via at least one pressure relief valve [(4) which is] connected downstream of the pressure control valve [(10)].

9. (Amended Once) The [P]process [according to] of Claim 8, [characterised in that] wherein [the holding pressure of] at least one of the pressure relief valves [(4) is] has a holding pressure equal to or higher than the pressure at which a critical propellant is held in the intermediate cycle times.

10. (Amended Once) The [P]process according to [one of the preceding claims, characterised in that] Claim 1 further comprising the step of regulating the pressure preset by the pressure control valve [(10) is regulated] via one or more pressure relief valves [(4)] to the injection pressure at which the propellant is added to the melt via an injection point [(5)].

11. (Amended Once) The [P]process [according to one of the preceding claims, characterised in that] of claim 1, wherein the injection point [(5)] is configured as a throttle means.

12. (Amended Once) The [P]process [according to] of Claim 11, [characterised in that] wherein the injection point [(5)] is in the form of a defined gap in an injector or of an injector with a sinter metal.

13. (Amended Once) The [P]process [according to one of Claims 11 or 12, characterised in that] of Claim 11, wherein the injection point [(5)] is configured as a controlled closure mechanism.

14. (Amended Once) The [P]process [according to] of Claim 1 [or one of the preceding Claims 3 to 13, characterised in that] further comprising the step of using water [is used] as the propellant.

15. (Amended Once) The [P]process [according to one of the preceding Claims 1 to 13, characterised in that] of Claim 1 further comprising the step of using a gas or gas mixture [is used] as the propellant.

16. (Amended Once) The [P]process [according to] of Claim[s] 15, [characterised in that] further comprising the step of using carbon dioxide [is used] as the propellant.

17. (Amended Once) The [P]process [according to] of Claim[s] 16, [characterised in that] wherein the carbon dioxide is held in the intermediate cycle times at a pressure of at least 60 bar [(= p (crit) CO₂ at room temperature)].

18. (Amended Once) The [P]process [according to one of the preceding claims, characterised in that for] of Claim 1 [the propellant injection phase] further comprising the step of elevating the

pressure of the propellant [is brought] during the propellant injection phase to a pressure of over 60 bar [via] using the pressure control valve [(10)].

19. (Amended Once) The [P]process [according to one of the preceding claims, characterised in that] of Claim 1 further comprising the step of generating a counterpressure [is generated] in the cavity [(1)].

20. (Amended Once) The [P]process [according to one of the preceding claims, characterised in that] of Claim 1, wherein the physically foamed injection [moulded] molded article is selected from the group consisting of a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a fender, a float and a closing means for bottle-like containers.

21. (Amended Once) A [D]device for the metered addition of physical propellants to a foamable melt, [wherein the device comprises] comprising:

a storage means [(11)], in which the propellant is stored under pressure,

a pressure control valve [(10)] for regulating the propellant pressure, and

an injection point [(5)], which is configured as a throttle means, at which the propellant under pressure is fed to the melt, [characterised in that]

wherein a controlled closure mechanism is provided at the injection point [(5)].

22. (Amended Once) The [D]device [for the metered addition of physical propellants according to] of Claim 21, [characterised in that instead of the controlled closure mechanism or in addition to the controlled closure mechanism,] further comprising at least one pressure relief valve [(4) is provided].

Marked up copy of
Substitute
specification

1 **Process for the Production of Physically Foamed Injection Moulded Articles**

2
3 The present invention

4 **PROCESS FOR THE PRODUCTION OF PHYSICALLY FOAMED**
5 **INJECTION MOLDED ARTICLES**

6
7 **BACKGROUND**

8 **Technical Field**

9 The present disclosure relates to a process for the production of physically
10 foamed injection moulded articles and, in particular, to a process for the production
11 of physically foamed injection moulded articles with an internal foam structure and
12 a compact closed-pore external skin of the same material as the base body.

13
14 **Related Art**

15 The production of foamed plastics, for example, is achieved with the aid of so-
16 called propellants, which expand a plastic, generally thermally softened plastic mass
17 in the desired manner. In this case, the propellants are either generated in situ via
18 chemical reaction of the components (chemical propellants), or compressed fluids, e.g.
19 N₂, CO₂, are added under pressure to the starting material, in which case a foaming
20 process of the plastic mass caused by the propellant is initiated upon the subsequent
21 drop in pressure of the component mixture to normal pressure.

22
23
24 However, chemical propellants have a series of disadvantages. For instance,
25 for use in foam injection moulding higher temperatures than are actually necessary for
26 softening starting materials may have to be selected in order to reach the ignition point
27 of the propellants, since the temperature at which the reaction of the components
28 generating propellant starts is generally very high. Because of the high temperatures
29 a higher expenditure of energy is necessary during melting of the raw materials. In
30 addition, the cycle or cooling times are increased and a higher cooling power of the

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cooling plants is necessary. In some circumstances, damage to the raw materials may also occur as a result of the comparatively high temperatures.

Chemical propellants which have not been converted can locate on the surface of the articles obtained and cause yellowing of the articles. Allergic skin reactions may also result upon contact with these articles.

Foam articles which have been obtained by means of chemical propellants are not recyclable, or if so only conditionally, since there is the risk that non-ignited propellants can lead to uncontrolled reactions during reuse.

Therefore, physical propellants are preferably used to foam plastics. Physical propellants allow optimum adaptation of the melting temperature to the respectively selected raw material, as a result of which the energy expenditure is reduced, optimum cycle and cooling times are made possible and, in addition, there is no risk that the raw materials could be detrimentally affected as a result of temperatures which are too high. Moreover, inexpensive gases such as CO₂, for example, can be used as physical propellants.

Physical propellants do not remain in the finished foam articles, but diffuse out within a comparatively short time. Therefore, these articles are fully recyclable, since there is no need to fear that propellant residues could lead to uncontrolled reactions.

Various processes are known for the production of articles from foamed plastic with a compact closed external skin and a cellular core cohering with the external skin or edge zone, also referred to as integral foam or structural foam.

For example, in the reaction injection moulding process (RIM), two reactive components are mixed together which harden and foam in the cavity of a mould under reaction. Because of the quicker cooling at the wall of the mould, the reaction mass solidifies more quickly there than in the interior of the mould, and, as a result, the foaming process ceases earlier there than in the mould interior, and a compact sealed external layer is formed.

As determined by the process, the reactive component mixture must be comparatively liquid in order to guarantee complete filling of the mould before the reaction starts. However, this leads to irregularities on the surface of the formed article as a result of spray over and skin formation, which necessitates expensive finishing for high-grade articles, for which a perfect surface is required.

Moreover, for the RIM process the mould must be treated with a separating agent prior to injection, which on the one hand requires more expenditure in processing and can additionally lead to residues on the finished article which must be removed. The relatively long cycle times are also disadvantageous.

Since

Because foaming in the RIM process is generally conducted chemically, the articles to be obtained are only conditionally recyclable.

Integral foams made of polyurethane to be used as working material primarily in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are preferably produced using the RIM process. However, for this field of application the

1 articles must not only have as perfect a surface as possible, but also have pleasant skin
2 feel (tactility).

3
4
5 It has been shown that articles of polyurethane integral foam have only a
6 conditionally acceptable tactility.

7
8
9 It is also known to produce integral foams from thermoplastic urethane or
10 thermoplastic elastomer by means of conventional injection moulding processes.
11 Both chemical and physical propellants can be used in this case. Contrary to the RIM
12 process, which requires special plants, already existing injection moulding plants
13 without expensive refitting can be used for this.

14
15 The necessary finishing of the articles obtained is only slight.

16
17 DE 196 46 665 A1 describes a process for metering physical propellants,
18 wherein a propellant is added at high pressure to the softened plastic material
19 transported in the consumer, e.g. an extruder or an RIM machine, and the amount of
20 propellant is regulated with a pressure control valve, which keeps the pressure
21 difference constant via a rigid throttle means by regulating the pressure difference in
22 dependence on the flow of propellant. The extrusion processes described here are
23 continuous processes in which the propellant is permanently added.

24
25 A process for the production of multilayered articles with a foamed core and
26 a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a
27 first propellant-free melt and a second melt containing propellant as well as possibly
28 a third propellant-free melt are firstly prepared and injected one after the other into an

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1 appropriate mould, in which case the mould must be maintained at a temperature
2 equal to or higher than the activation temperature of the propellant.

3
4
5 Where physical propellants are used, it is suggested that either the selected
6 temperature of the melt upon leaving the nozzle is so high that, when a mould with
7 constant internal volume is used, the gas formation, and thus the expansion, still
8 occurs below the pressure exerted on the substance in the mould, and when a mould
9 with extendable interior is used, the gas formation, and thus the expansion, occurs by
10 relieving the pressure exerted on the mould interior to expand the mould. There is no
11 mention of the propellant being added directly to the melt flow which flows into the
12 mould, nor of the quantity of propellant apportioned to the melt flow being regulated
13 via the pressure.

14
15
16 An improved process of the aforementioned type is specified in DE 1 948 454,
17 wherein the propellant is injected into the melt flow shortly before entry into the
18 mould and the injection period is continued until the mixture quantity required to form
19 the core has been inserted into the mould. Solvents with a boiling point preferably
20 between 20 and 150°C are specified as propellants, which are to prevent premature
21 expansion under a corresponding pressure. There is likewise no mention here of a
22 pressure regulation of the added quantity of propellant to the melt.

23
24
25 A process for the production of injection moulded articles with foamed core
26 is described in the US Patent No. 4,548,776, according to which gaseous or gas-
27 generated chemical propellant is already added to the melt in the extruder, is
28 thoroughly mixed with this and the already foamed melt is then injected into the
29 mould.

In this case, the addition of propellant occurs via a porous insert at the injection point, a supply valve being provided in the feed pipe. This supply valve can be connected to an automatic control device, via which the pressure of the propellant to be fed is adjusted.

The object of the present invention is to provide a process for the production of physically foamed injection moulded articles, with which injection moulded articles with an integral structure, excellent surface characteristics, thus rendering expensive finishing unnecessary, and additionally excellent tactility, can be obtained in a simple manner using conventional injection moulding plants, thus rendering expensive finishing unnecessary.

SUMMARY

The articles produced according to the invention are suitable in particular for fields of application which set high quality requirements for surface structure and for which a pleasant sensory feel is of advantage on skin contact. The automobile industry is given as an example, for which handles, knobs such as gearshift knobs, steering wheel casings etc. of the foamed plastics obtained according to the invention can be used. However, the process according to the invention is in no way restricted to the production of articles for the automobile industry, but is quite generally suitable for the production of any desired foamed injection moulded articles.

For example, mass-produced articles such as closing means for bottle-like containers, e.g. stoppers or corks, may also be advantageously obtained according to this process. Further examples are balls, spheres, fenders, floats, etc.

1 A further field of use is the production of supporting parts, for example, for
2 the aviation or automobile industry, in particular for parts where strength is relevant.

3
4
5 This object is achieved according to the ~~inventiondisclosure~~ by a process for
6 the production of physically foamed injection moulded articles, wherein firstly in a
7 first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a
8 second stage a physical propellant is added at elevated pressure to the following melt
9 portion (propellant injection phase), wherein metering of the physical propellant
10 occurs at least in a pressure regulated manner, wherein the pressure which is exerted
11 on the propellant during the propellant injection phase is greater than the pressure
12 which is exerted on the propellant in the phases between or before or after metered
13 addition, and the expansion of the propellant occurs in the cavity, and possibly in a
14 third stage a propellant-free further melt portion is charged into the cavity.

15
16
17 This process also permits the formation of physically foamed injection
18 moulded articles, the foamed core of which is completely or partially enclosed by a
19 compact closed external skin, which has been produced without the addition of
20 propellants, the core and the external skin being made of the same material.

21
22
23 The present ~~inventiondisclosure~~ additionally relates to a device for the metered
24 addition of propellants under elevated pressure to an expandable melt.

25
26 This device can also be advantageously used for the metered addition of
27 compressible propellants.

28

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1 The propellant-free melt portion firstly fed into the cavity in the first stage
2 forms a compact closed external skin without pores in the finished foamed injection
3 moulded articles.

4
5 Any desired fluid which expands upon corresponding pressure relief and
6 foams the melt material in a suitable manner can be used as propellant. Hence,
7 compressible fluids such as gases in liquid or supercritical phase, for example, may
8 be used.

9
10 The use of carbon dioxide is recommended because of its ready availability.

11
12 A further preferred propellant is water.

13
14 The starting material for the melt is not subject to any special restrictions. Any
15 desired thermoplastic melt material which is suitable for injection moulding and can
16 be foamed may be used.

17
18 Examples are thermoplastic materials, but also further thermoplastic melts,
19 such as metallic or ceramic melts, for example. Examples of metallic materials
20 include aluminium, magnesium, zinc, tin or even precious metals.

21
22 The process according to the invention leads to weight reduction and
23 strength increase in comparison to the corresponding compact articles.

24
25 "Pressure regulated" in the sense of the invention means that in the
26 course of the process the pressure exerted on the propellant is varied for metered
27 addition of the propellant.

28

In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve.

Therefore, according to the invention disclosure, the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure.

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present invention disclosure is explained in more detail below with reference to the figures on the basis of a preferred embodiment by the example of the addition of a compressible fluid. It goes without saying that the following explanation may also be applied in principle to non-compressible fluids such as water, for example.

Figures 1a-1d

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other objects and advantages of the embodiments described herein will become apparent with reference to the following detailed description when taken in conjunction with the accompanying drawings in which:

FIGS. 1A-1D show the individual stages of the process
 according to the invention disclosure for the production
 of physically foamed injection moulded
 articles;

Figure

FIG. 2 schematically shows a device for executing the
 process according to the invention;

Figure disclosure;

FIG. 3 is a graph showing the pressure curve during
 execution of the process;

Figure and

FIG. 4 shows a variant of Figure FIG. 1 with direct
 introduction of the propellant into the cavity.

DETAILED DESCRIPTION OF THE DRAWINGS

As Figure FIG. 1aA shows, the cavity 1 of any injection moulding plant is
 partially initially filled in a first stage firstly with a compact propellant-free melt 6.
 In this case, the feed pipe 3 for a compressed propellant is closed, for example, by
 a valve 4 such as a pressure relief valve 4 (overflow valve).

After the cavity 1 has been filled with a desired quantity of propellant-free
 melt 6, the feed pipe 3 for the propellant is opened and the propellant is injected in
 compressed, preferably liquid, state via the injection point 5. Through contact with
 the hot melt, the liquid propellant turns to gas and expands under the lower pressure
 in the cavity.

As a general rule the propellant is still liquid and not gaseous at the injection point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower sense.

The

A mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes the cavity 1 to fill completely, in which case the propellant-free melt portion 6 which was used for the initial filling comes to rest in the region of the cavity walls and forms the external skin or edge zone of the injection moulded article to be formed.

The cavity 1 can be ready filled as desired and required up to the maximum filling quantity with melt mixed with propellant or, as shown in Figure FIG. 1dD, propellant-free melt can again be fed to the cavity in a third stage.³⁴ In this case a foamed article is obtained which has a compact firm external skin right around which is formed by propellant-free melt.

After foaming and hardening, the finished injection moulded article, e.g. made of integral foam, is removed from the cavity and the cavity is immediately available again for the next charge.

As shown in Figure FIG. 1dD, injection moulded articles, which have a cellular foamed internal core and a compact firm closed external skin, are obtained with the process according to the invention disclosure.

Contrary to the known foaming processes, such as those described above, in which the cavity is filled completely with a melt/propellant mixture, according to the

inventiondisclosure an initial filling with propellant-free melt occurs firstly, as a result of which the formation of a uniform closed compact external skin is effected and articles with excellent surface characteristics can be obtained.

It is essential for execution of the process to prevent premature expansion of the propellant held under pressure. This can be achieved by appropriate insulation of the device and/or maintaining a suitable pressure level.

The metered addition of the propellant is conducted in a time- and pressure-controlled manner for the process according to the inventiondisclosure. Control can be carried out via a device which is also the subject of the inventiondisclosure.

As shown in FigureFIG 2, the propellant stored under pressure in a storage means 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a multi-way valve such as a 3/3- or 2/3-way proportional valve, and should advantageously have a very quick reaction time and precise regulation.

During the propellant injection phase, i.e. the phase in which the propellant is added to the melt, in the case of critical propellants, the compressed propellant passes via a pressure relief valve 4 to the injection point 5 and there is added to the melt.

In this case, the dimensions of the pipes, connection pieces and also the parts of the technical control system of the process are such that no premature expansion in volume of the propellant under pressure is possible.

1 In the case of a sudden increase in volume the aggregate state of the agent can
2 change, i.e. the agent changes into a gas, in which case vaporization cold is
3 generated, which would in turn block the pipes as a result of "icing".
4

5
6 An increase in temperature on the way to the injection point 5 would also lead
7 to a change in the aggregate state. For prevention, insulation of the heat-carrying
8 elements is recommended.
9

10
11 In order to prevent premature expansion, all feed pipes should be as short as
12 possible. Consequently, the pressure control valve 10 is preferably constructed to be
13 as close as possible to the injection point 5. An improvement to the control
14 characteristics of the control valve is also achieved as a result of the thus shortened
15 feed pipe to the injection point 5.
16

17
18 If critical propellants are used, a pressure relief valve or overflow valve 4 is
19 provided before the injection point 5, this valve ensuring that the pressure in the
20 device does not drop below a specific value, preferably p_{crit} at the given
21 temperature, at which the transformation of the propellant into gas would take place.
22 If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar
23 should be maintained at room temperature in order to keep the carbon dioxide in the
24 device upstream in liquid state.
25

26
27 The pressure relief valve 4 ensures that the propellant remains in compressed
28 state even during outage times of the machine, e.g. in the intermediate cycle times
29 before and after or between the propellant injection phases. A full release of pressure

only occurs when the machine or control system is switched off. Several pressure relief valves with "falling" pressure values may also be provided so that a pressure gradient is formed in front of the injection point 5 in the feed pipe section between the pressure control valve 10 and the pressure relief valve 4.

The graph in Figure FIG. 3 schematically shows the pressure curve for executing the process according to the invention using the example of compressible propellants.

Outside of the propellant injection phase, as in the intermediate cycle times, it is sufficient to keep the device at a selected pressure, at which the propellant respectively used remains in compressed, preferably liquid, state (section 20).

During the propellant injection phase (section 22), an elevated pressure is introduced in the feed pipes through the pressure control valve 10 so that the opening pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section 3 up to the injection point 5 is quickly filled with liquid medium.

In this case, the pressure increase is proportional to the desired quantity of propellant to be fed to the melt. After time t expires, as soon as the desired quantity of propellant has been added to the melt, the pressure is reduced again to the starting pressure (section 24).

In Figure FIG. 3, sections 21 and 23 show the pressure build up or reduction phase.

1 The injection point 5 is preferably configured as a throttle means, e.g. as a
2 defined gap in an injector, a sintered metal injector, or a needle valve. According to
3 the invention disclosure, a controlled closure mechanism is located at the injection
4 point. The quick pressure increase and the resistance through the injector prevent the
5 propellant from transforming into gas, while the agent flows on from the pressure
6 control valve 10.

7
8
9 The above measures ensure that the transformation of the agent into gas only
10 occurs upon exit from the injector and when in contact with the hot melt, and that the
11 inflowing melt is foamed.

12
13
14 The controlled closure mechanism can be omitted if a pressure relief valve is
15 provided.

16
17
18 After the propellant injection phase has ended, i.e. after the desired quantity
19 of propellant has been added to the melt, the pressure in the feed pipe to the injection
20 point 5 is reduced so that no propellant can flow on. However, in the pipe up to the
21 pressure relief valve 4 the starting pressure remains in order to keep the agent in
22 compressed or liquid state for the next cycle. A virtually pressure-free and thus
23 gaseous state prevails only in the short feed pipe section from the pressure relief valve
24 4 to the injection point 5 until the next cycle.

25
26
27 It goes without saying that this part of the plant may also be kept under
28 pressure if required by the provision of a suitable closure mechanism which opens

again at the beginning of the propellant injection phase as a result of the increasing pressure level.

The pressure control via the pressure control valve can occur automatically by providing pressure measurement points 12, 13, for example, in front of and behind the pressure control valve.

If carbon dioxide is used as propellant, for example, the plant is preferably held at an operating pressure of at least 60 bar at room temperature, so that the CO₂ also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure of about 200 bar, for example, is built up (section 21) in order to assure an adequate flow of propellant to the melt. After the propellant injection phase 22 has ended, the pressure is reduced again to the desired operating pressure.

The injection point 5 is preferably located in the feeder pipe 3 close to the spray point ~~x~~"x". According to a further embodiment, as is shown in Figure FIG. 4, the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity.

In addition, the build up of a counterpressure can be provided in the cavity 1, such as is also used in conventional injection moulding processes in the so-called gas counterpressure process.

Very short cycle times can be obtained with the process according to the invention. Hence, the process according to the invention is also

1 very well suited to the production of mass-produced articles. The short cycle times
2 are supported by the vaporization cold resulting upon the transformation of the
3 propellant into gas, and this causes a reduction in the cooling time, and thus also the
4 cycle time.

5
6
7 Should there still be propellant residues present in the pore structure in the
8 core of the article after demoulding, these slowly diffuse out of the article without
9 detriment to its usability or recyclability.

10
11
12 Excellent dimensional stability of the article is achieved as a result of its
13 closed firm external skin. In addition, foamed injection moulded articles which have
14 a homogeneous uniform external skin and excellent tactility can be obtained with the
15 process according to the invention disclosure.

16
17
18 The foamed injection moulded articles obtained have an excellent surface
19 quality and do not require any further finishing. It is also of advantage that the cavity
20 does not need to be treated with a separating agent.

21
22
23 The process according to the invention disclosure for the pressure-controlled
24 metered addition of physical propellants to an expandable melt can be conducted
25 advantageously with a device comprising a storage means 11, in which the propellant
26 is stored under pressure, a pressure control valve 10 for regulating the propellant
27 pressure and an injection point 5, which is preferably configured as a throttle means,
28 at which the propellant under pressure is added to the melt, wherein the injection point
29 5 includes a controlled closure mechanism, and in the case of critical propellants at

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1 least one pressure relief valve 4 is provided which is positioned downstream of the
2 pressure control valve 10.

3

4

5 Although the above-described process and the device for the pressure-
6 controlled metered addition of propellants under high pressure can be advantageously
7 used for the production of physically foamed injection moulded articles, they are, of
8 course, also suitable for other processes in which propellants are added under high
9 pressure to melts to be expanded.

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1 **List of Reference Numbers**

2

3

4 1 ————— cavity

5 2 ————— melt feed

6 3 ————— propellant feed pipe

7 4 ————— pressure relief valve

8 5 ————— injection point

9 6 ————— propellant-free melt

10 7 ————— melt with added propellant

11 8 ————— injection of plastic material

12 9 ————— mould comprising two halves

13 10 ————— pressure control valve

14 11 ————— propellant storage means

15

16 x ————— spray point

17

18 Section 20 ————— pressure during the intermediate cycle-
19 times

20 Section 21 ————— pressure build up phase

21 Section 22 ————— propellant injection phase

22 Section 23 ————— pressure reduction phase

23

24

25

26

27

28

29

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1 **Claims:**

2

3 1. ~~Process for the production of~~

4 ~~What is claimed is:~~

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PROCESS FOR THE PRODUCTION OF PHYSICALLY FOAMED
INJECTION MOLDED ARTICLES

ABSTRACT

A process for producing physically foamed injection moulded articles; wherein in a first stage is provided. The process involves feeding a propellant-free first melt portion (6) is fed into a cavity (1) (initial filling), in a second stage thermoplastic melt into a cavity followed by delivering a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), and possibly in a third stage a propellant-free further melt portion is charged directly into the cavity (1), the production of the injection moulded articles occurring in the cavity, characterised in that metering of the physical propellant in the second stage occurs in a pressure regulated manner, wherein the pressure which is or directly into the melt flowing into the cavity. The pressure exerted on the propellant during the propellant injection phase stage is greater than the pressure which is that exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity (1).

2. — Process according to Claim 1, characterised in that the propellant is a compressible fluid.

3. — Process according to Claim 1 or 2, characterised in that the propellant is kept under pressure in the intermediate cycle times before and after the propellant injection phase, or is present in a compressed state.

4. — Process according to Claim 3, characterised in that in the intermediate cycle times the propellant is held a pressure of at least p_{crit} of the propellant at the given temperature.

1

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1 ~~5. Process according to one of the preceding claims,~~
2 ~~characterised in that the pressure exerted on the~~
3 ~~propellant is controlled via a pressure control valve (10).~~

4
5 ~~6. Process according to Claim 5, characterised in that~~
6 ~~the pressure control valve (10) is a multi-way valve.~~

7
8 ~~7. Process according to Claim 6, characterised in that~~
9 ~~a 3/3-way proportional valve or a 2/3-way proportional valve is used as multi-way~~
10 ~~valve.~~

11
12 ~~8. Process according to one of the preceding claims, characterised in that the~~
13 ~~pressure control in the case of critical propellants additionally occurs via at least~~
14 ~~one pressure relief valve (4) which is connected downstream of the pressure~~
15 ~~control valve (10).~~

16
17 ~~9. Process according to Claim 8, characterised in that~~
18 ~~the holding pressure of at least one of the pressure relief valves (4) is equal to or~~
19 ~~higher than the pressure at which a critical propellant is held in the intermediate~~
20 ~~cycle times.~~

21
22 ~~10. Process according to one of the preceding claims, characterised in that the~~
23 ~~pressure preset by the pressure control valve (10) is regulated via one or more~~
24 ~~pressure relief valves (4) to the injection pressure at which the propellant is added~~
25 ~~to the melt via an injection point (5).~~

26
27 ~~11. Process according to one of the preceding claims, characterised in that the~~
28 ~~injection point (5) is configured as a throttle means.~~

29

- 1 ~~12. — Process according to Claim 11, characterised in that~~
2 ~~the injection point (5) is in the form of a defined gap in an injector or of an injector~~
3 ~~with a sinter metal.~~
4
- 5 ~~13. — Process according to one of Claims 11 or 12,~~
6 ~~characterised in that the injection point (5) is configured as a controlled closure~~
7 ~~mechanism.~~
8
- 9 ~~14. — Process according to Claim 1 or one of the preceding Claims 3 to 13,~~
10 ~~characterised in that water is used as propellant.~~
11
- 12 ~~15. — Process according to one of the preceding Claims 1 to 13, characterised in~~
13 ~~that a gas or gas mixture is used as propellant.~~
14
- 15 ~~16. — Process according to Claims 15, characterised in that~~
16 ~~carbon dioxide is used as propellant.~~
17
- 18 ~~17. — Process according to Claims 16, characterised in that~~
19 ~~the carbon dioxide is held in the intermediate cycle times at a pressure of at least~~
20 ~~60 bar (= p_{crit} CO₂ at room temperature).~~
21
- 22 ~~18. — Process according to one of the preceding claims, characterised in that for~~
23 ~~the propellant injection phase the propellant is brought to a pressure of over 60 bar~~
24 ~~via the pressure control valve (10).~~
25
- 26 ~~19. — Process according to one of the preceding claims, characterised in that a~~
27 ~~counterpressure is generated in the cavity (1).~~
28
- 29 ~~20. — Process according to one of the preceding claims,~~

1 characterised in that the physically foamed injection moulded article is selected
2 from a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a
3 fender, a float and a closing means for bottle-like containers.

4
5 21. — Device for the metered addition of physical propellants to a foamable melt,
6 wherein the device comprises a storage means (11), in which the propellant is
7 stored under pressure, a pressure control valve (10) for regulating the propellant
8 pressure, and an injection point (5), which is configured as a throttle means, at
9 which the propellant under pressure is fed to the melt,
10 characterised in that a controlled closure mechanism is provided at the injection
11 point (5):

12
13 22. — Device for the metered addition of physical propellants according to Claim
14 21, characterised in that instead of the controlled closure mechanism or in addition
15 to the controlled closure mechanism, at least one pressure relief valve (4) is
16 provided:

17
18 during the holding phase.
19

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1 PROCESS FOR THE PRODUCTION OF PHYSICALLY FOAMED
2 INJECTION MOLDED ARTICLES

3
4 BACKGROUND

5 Technical Field

6 The present disclosure relates to a process for the production of physically
7 foamed injection molded articles and, in particular, to a process for the production of
8 physically foamed injection molded articles with an internal foam structure and a
9 compact closed-pore external skin of the same material as the base body.

10
11 Related Art

12 The production of foamed plastics, for example, is achieved with the aid of so-
13 called propellants, which expand a plastic, generally thermally softened plastic mass
14 in the desired manner. In this case, the propellants are either generated in situ via
15 chemical reaction of the components (chemical propellants), or compressed fluids, e.g.
16 N₂, CO₂, are added under pressure to the starting material, in which case a foaming
17 process of the plastic mass caused by the propellant is initiated upon the subsequent
18 drop in pressure of the component mixture to normal pressure.

19 However, chemical propellants have a series of disadvantages. For instance,
20 for use in foam injection molding higher temperatures than are actually necessary for
21 softening starting materials may have to be selected in order to reach the ignition point
22 of the propellants, since the temperature at which the reaction of the components
23 generating propellant starts is generally very high. Because of the high temperatures
24 a higher expenditure of energy is necessary during melting of the raw materials. In
25 addition, the cycle or cooling times are increased and a higher cooling power of the
26 cooling plants is necessary. In some circumstances, damage to the raw materials may
27 also occur as a result of the comparatively high temperatures.

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1 Chemical propellants which have not been converted can locate on the surface
2 of the articles obtained and cause yellowing of the articles. Allergic skin reactions
3 may also result upon contact with these articles.

4 Foam articles which have been obtained by means of chemical propellants are
5 not recyclable, or if so only conditionally, since there is the risk that non-ignited
6 propellants can lead to uncontrolled reactions during reuse.

7 Therefore, physical propellants are preferably used to foam plastics. Physical
8 propellants allow optimum adaptation of the melting temperature to the respectively
9 selected raw material, as a result of which the energy expenditure is reduced, optimum
10 cycle and cooling times are made possible and, in addition, there is no risk that the
11 raw materials could be detrimentally affected as a result of temperatures which are too
12 high. Moreover, inexpensive gases such as CO₂, for example, can be used as physical
13 propellants.

14 Physical propellants do not remain in the finished foam articles, but diffuse out
15 within a comparatively short time. Therefore, these articles are fully recyclable, since
16 there is no need to fear that propellant residues could lead to uncontrolled reactions.

17 Various processes are known for the production of articles from foamed plastic
18 with a compact closed external skin and a cellular core cohering with the external skin
19 or edge zone, also referred to as integral foam or structural foam.

20 For example, in the reaction injection molding process (RIM), two reactive
21 components are mixed together which harden and foam in the cavity of a mold under
22 reaction. Because of the quicker cooling at the wall of the mold, the reaction mass
23 solidifies more quickly there than in the interior of the mold and, as a result, the
24 foaming process ceases earlier there than in the mold interior, and a compact sealed
25 external layer is formed.

26 As determined by the process, the reactive component mixture must be
27 comparatively liquid in order to guarantee complete filling of the mold before the
28 reaction starts. However, this leads to irregularities on the surface of the formed

1 article as a result of spray over and skin formation, which necessitates expensive
2 finishing for high-grade articles, for which a perfect surface is required.

3 Moreover, for the RIM process the mold must be treated with a separating
4 agent prior to injection, which on the one hand requires more expenditure in
5 processing and can additionally lead to residues on the finished article which must be
6 removed. The relatively long cycle times are also disadvantageous.

7 Because foaming in the RIM process is generally conducted chemically, the
8 articles to be obtained are only conditionally recyclable.

9 Integral foams made of polyurethane to be used as working material primarily
10 in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are
11 preferably produced using the RIM process. However, for this field of application the
12 articles must not only have as perfect a surface as possible, but also have pleasant skin
13 feel (tactility).

14 It has been shown that articles of polyurethane integral foam have only a
15 conditionally acceptable tactility.

16 It is also known to produce integral foams from thermoplastic urethane or
17 thermoplastic elastomer by means of conventional injection molding processes. Both
18 chemical and physical propellants can be used in this case. Contrary to the RIM
19 process, which requires special plants, already existing injection molding plants
20 without expensive refitting can be used for this.

21 The necessary finishing of the articles obtained is only slight.

22 DE 196 46 665 A1 describes a process for metering physical propellants,
23 wherein a propellant is added at high pressure to the softened plastic material
24 transported in the consumer, e.g. an extruder or an RIM machine, and the amount of
25 propellant is regulated with a pressure control valve, which keeps the pressure
26 difference constant via a rigid throttle means by regulating the pressure difference in
27 dependence on the flow of propellant. The extrusion processes described here are
28 continuous processes in which the propellant is permanently added.

1 A process for the production of multilayered articles with a foamed core and
2 a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a
3 first propellant-free melt and a second melt containing propellant as well as possibly
4 a third propellant-free melt are firstly prepared and injected one after the other into an
5 appropriate mold, in which case the mold must be maintained at a temperature equal
6 to or higher than the activation temperature of the propellant.

7 Where physical propellants are used, it is suggested that either the selected
8 temperature of the melt upon leaving the nozzle is so high that, when a mold with
9 constant internal volume is used, the gas formation, and thus the expansion, still
10 occurs below the pressure exerted on the substance in the mold, and when a mold with
11 extendable interior is used, the gas formation, and thus the expansion, occurs by
12 relieving the pressure exerted on the mold interior to expand the mold. There is no
13 mention of the propellant being added directly to the melt flow which flows into the
14 mold, nor of the quantity of propellant apportioned to the melt flow being regulated
15 via the pressure.

16 An improved process of the aforementioned type is specified in DE 1 948 454,
17 wherein the propellant is injected into the melt flow shortly before entry into the mold
18 and the injection period is continued until the mixture quantity required to form the
19 core has been inserted into the mold. Solvents with a boiling point preferably between
20 20 and 150°C are specified as propellants, which are to prevent premature expansion
21 under a corresponding pressure. There is likewise no mention here of a pressure
22 regulation of the added quantity of propellant to the melt.

23 A process for the production of injection molded articles with foamed core is
24 described in U.S. Patent No. 4,548,776, according to which gaseous or gas-generated
25 chemical propellant is already added to the melt in the extruder, is thoroughly mixed
26 with this and the already foamed melt is then injected into the mold.

27 In this case, the addition of propellant occurs via a porous insert at the
28 injection point, a supply valve being provided in the feed pipe. This supply valve can

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1 be connected to an automatic control device, via which the pressure of the propellant
2 to be fed is adjusted.

3 The object of the present disclosure is to provide a process for the production
4 of physically foamed injection molded articles, with which injection molded articles
5 with an integral structure, excellent surface characteristics, and excellent tactility, can
6 be obtained in a simple manner using conventional injection molding plants, thus
7 rendering expensive finishing unnecessary.

8 9 SUMMARY

10 The articles produced according to the disclosure are suitable in particular for
11 fields of application which set high quality requirements for surface structure and for
12 which a pleasant sensory feel is of advantage on skin contact. The automobile
13 industry is given as an example, for which handles, knobs such as gearshift knobs,
14 steering wheel casings etc. of the foamed plastics obtained according to the disclosure
15 can be used. However, the process according to the disclosure is in no way restricted
16 to the production of articles for the automobile industry, but is quite generally suitable
17 for the production of any desired foamed injection molded articles.

18 For example, mass-produced articles such as closing means for bottle-like
19 containers, e.g. stoppers or corks, may also be advantageously obtained according to
20 this process. Further examples are balls, spheres, fenders, floats, etc.

21 A further field of use is the production of supporting parts, for example, for
22 the aviation or automobile industry, in particular for parts where strength is relevant.

23 This object is achieved according to the disclosure by a process for the
24 production of physically foamed injection molded articles, wherein firstly in a first
25 stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second
26 stage a physical propellant is added at elevated pressure to the following melt portion
27 (propellant injection phase), wherein metering of the physical propellant occurs at
28 least in a pressure regulated manner, wherein the pressure which is exerted on the
29 propellant during the propellant injection phase is greater than the pressure which is

1 exerted on the propellant in the phases between or before or after metered addition,
2 and the expansion of the propellant occurs in the cavity, and possibly in a third stage
3 a propellant-free further melt portion is charged into the cavity.

4 This process also permits the formation of physically foamed injection molded
5 articles, the foamed core of which is completely or partially enclosed by a compact
6 closed external skin, which has been produced without the addition of propellants, the
7 core and the external skin being made of the same material.

8 The present disclosure additionally relates to a device for the metered addition
9 of propellants under elevated pressure to an expandable melt.

10 This device can also be advantageously used for the metered addition of
11 compressible propellants.

12 The propellant-free melt portion firstly fed into the cavity in the first stage
13 forms a compact closed external skin without pores in the finished foamed injection
14 molded articles.

15 Any desired fluid which expands upon corresponding pressure relief and
16 foams the melt material in a suitable manner can be used as propellant. Hence,
17 compressible fluids such as gases in liquid or supercritical phase, for example, may
18 be used.

19 The use of carbon dioxide is recommended because of its ready availability.

20 A further preferred propellant is water.

21 The starting material for the melt is not subject to any special restrictions. Any
22 desired thermoplastic melt material which is suitable for injection molding and can
23 be foamed may be used.

24 Examples are thermoplastic materials, but also further thermoplastic melts,
25 such as metallic or ceramic melts, for example. Examples of metallic materials
26 include aluminum, magnesium, zinc, tin or even precious metals.

27 The process according to the disclosure leads to weight reduction and strength
28 increase in comparison to the corresponding compact articles.

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"Pressure regulated" in the sense of the disclosure means that in the course of the process the pressure exerted on the propellant is varied for metered addition of the propellant. In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve.

Therefore, according to the disclosure, the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure.

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present disclosure is explained in more detail below with reference to the figures on the basis of a preferred embodiment by the example of the addition of a compressible fluid. It goes without saying that the following explanation may also be applied in principle to non-compressible fluids such as water, for example.

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other objects and advantages of the embodiments described herein will become apparent with reference to the following detailed description when taken in conjunction with the accompanying drawings in which:

FIGS. 1A-1D show the individual stages of the process according to the disclosure for the production of physically foamed injection molded articles;

FIG. 2 schematically shows a device for executing the process according to the disclosure;

FIG. 3 is a graph showing the pressure curve during execution of the process;
and

FIG. 4 shows a variant of FIG. 1 with direct introduction of the propellant into
the cavity.

DETAILED DESCRIPTION OF THE DRAWINGS

As FIG. 1A shows, the cavity 1 of any injection molding plant is partially
initially filled in a first stage firstly with a compact propellant-free melt 6. In this
case, a feed pipe 3 for a compressed propellant is closed, for example, by a valve 4
such as a pressure relief valve 4 (overflow valve).

After the cavity 1 has been filled with a desired quantity of propellant-free
melt 6, the feed pipe 3 for the propellant is opened and the propellant is injected in
compressed, preferably liquid, state via the injection point 5. Through contact with
the hot melt, the liquid propellant turns to gas and expands under the lower pressure
in the cavity.

As a general rule the propellant is still liquid and not gaseous at the injection
point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower
sense.

A mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes
the cavity 1 to fill completely, in which case the propellant-free melt portion 6 which
was used for the initial filling comes to rest in the region of the cavity walls and forms
the external skin or edge zone of the injection molded article to be formed.

The cavity 1 can be ready filled as desired and required up to the maximum
filling quantity with melt mixed with propellant or, as shown in FIG. 1D, propellant-
free melt can again be fed to the cavity in a third stage. In this case a foamed article
is obtained which has a compact firm external skin right around which is formed by
propellant-free melt.

1 After foaming and hardening, the finished injection molded article, e.g. made
2 of integral foam, is removed from the cavity and the cavity is immediately available
3 again for the next charge.

4 As shown in FIG. 1D, injection molded articles, which have a cellular foamed
5 internal core and a compact firm closed external skin, are obtained with the process
6 according to the disclosure.

7 Contrary to the known foaming processes, such as those described above, in
8 which the cavity is filled completely with a melt/propellant mixture, according to the
9 disclosure an initial filling with propellant-free melt occurs firstly, as a result of which
10 the formation of a uniform closed compact external skin is effected and articles with
11 excellent surface characteristics can be obtained.

12 It is essential for execution of the process to prevent premature expansion of
13 the propellant held under pressure. This can be achieved by appropriate insulation of
14 the device and/or maintaining a suitable pressure level.

15 The metered addition of the propellant is conducted in a time- and pressure-
16 controlled manner for the process according to the disclosure. Control can be carried
17 out via a device which is also the subject of the disclosure.

18 As shown in FIG. 2, the propellant stored under pressure in a storage means
19 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a
20 multi-way valve such as a 3/3- or 2/3-way proportional valve, and should
21 advantageously have a very quick reaction time and precise regulation.

22 During the propellant injection phase, i.e. the phase in which the propellant is
23 added to the melt, in the case of critical propellants, the compressed propellant passes
24 via a pressure relief valve 4 to the injection point 5 and there is added to the melt.

25 In this case, the dimensions of the pipes, connection pieces and also the parts
26 of the technical control system of the process are such that no premature expansion
27 in volume of the propellant under pressure is possible.

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1 In the case of a sudden increase in volume the aggregate state of the agent can
2 change, i.e. the agent changes into a gas, in which case vaporization cold is generated,
3 which would in turn block the pipes as a result of "icing."

4 An increase in temperature on the way to the injection point 5 would also lead
5 to a change in the aggregate state. For prevention, insulation of the heat-carrying
6 elements is recommended.

7 In order to prevent premature expansion, all feed pipes should be as short as
8 possible. Consequently, the pressure control valve 10 is preferably constructed to be
9 as close as possible to the injection point 5. An improvement to the control
10 characteristics of the control valve is also achieved as a result of the thus shortened
11 feed pipe to the injection point 5.

12 If critical propellants are used, a pressure relief valve or overflow valve 4 is
13 provided before the injection point 5, this valve ensuring that the pressure in the
14 device does not drop below a specific value, preferably p_{crit} at the given
15 temperature, at which the transformation of the propellant into gas would take place.
16 If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar
17 should be maintained at room temperature in order to keep the carbon dioxide in the
18 device upstream in liquid state.

19 The pressure relief valve 4 ensures that the propellant remains in compressed
20 state even during outage times of the machine, e.g. in the intermediate cycle times
21 before and after or between the propellant injection phases. A full release of pressure
22 only occurs when the machine or control system is switched off. Several pressure
23 relief valves with "falling" pressure values may also be provided so that a pressure
24 gradient is formed in front of the injection point 5 in the feed pipe section between the
25 pressure control valve 10 and the pressure relief valve 4.

26 The graph in FIG. 3 schematically shows the pressure curve for executing the
27 process according to the disclosure using the example of compressible propellants.

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1 Outside of the propellant injection phase, as in the intermediate cycle times,
2 it is sufficient to keep the device at a selected pressure, at which the propellant
3 respectively used remains in compressed, preferably liquid, state (section 20).

4 During the propellant injection phase (section 22), an elevated pressure is
5 introduced in the feed pipes through the pressure control valve 10 so that the opening
6 pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section
7 3 up to the injection point 5 is quickly filled with liquid medium.

8 In this case, the pressure increase is proportional to the desired quantity of
9 propellant to be fed to the melt. After time "t" expires, as soon as the desired quantity
10 of propellant has been added to the melt, the pressure is reduced again to the starting
11 pressure (section 24).

12 In FIG. 3, sections 21 and 23 show the pressure build up or reduction phase.

13 The injection point 5 is preferably configured as a throttle means, e.g. as a
14 defined gap in an injector, a sintered metal injector, or a needle valve. According to
15 the disclosure, a controlled closure mechanism is located at the injection point. The
16 quick pressure increase and the resistance through the injector prevent the propellant
17 from transforming into gas, while the agent flows on from the pressure control valve
18 10.

19 The above measures ensure that the transformation of the agent into gas only
20 occurs upon exit from the injector and when in contact with the hot melt, and that the
21 inflowing melt is foamed.

22 The controlled closure mechanism can be omitted if a pressure relief valve is
23 provided.

24 After the propellant injection phase has ended, i.e. after the desired quantity
25 of propellant has been added to the melt, the pressure in the feed pipe to the injection
26 point 5 is reduced so that no propellant can flow on. However, in the pipe up to the
27 pressure relief valve 4 the starting pressure remains in order to keep the agent in
28 compressed or liquid state for the next cycle. A virtually pressure-free and thus

gaseous state prevails only in the short feed pipe section from the pressure relief valve 4 to the injection point 5 until the next cycle.

It goes without saying that this part of the plant may also be kept under pressure if required by the provision of a suitable closure mechanism which opens again at the beginning of the propellant injection phase as a result of the increasing pressure level.

The pressure control via the pressure control valve can occur automatically by providing pressure measurement points 12, 13, for example, in front of and behind the pressure control valve.

If carbon dioxide is used as propellant, for example, the plant is preferably held at an operating pressure of at least 60 bar at room temperature, so that the CO₂ also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure of about 200 bar, for example, is built up (section 21) in order to assure an adequate flow of propellant to the melt. After the propellant injection phase 22 has ended, the pressure is reduced again to the desired operating pressure.

The injection point 5 is preferably located in the feeder pipe 3 close to the spray point "x." According to a further embodiment, as is shown in FIG. 4, the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity.

In addition, the build up of a counterpressure can be provided in the cavity 1, such as is also used in conventional injection molding processes in the so-called gas counterpressure process.

Very short cycle times can be obtained with the process according to the disclosure. Hence, the process according to the disclosure is also very well suited to the production of mass-produced articles. The short cycle times are supported by the vaporization cold resulting upon the transformation of the propellant into gas, and this causes a reduction in the cooling time, and thus also the cycle time.

1 Should there still be propellant residues present in the pore structure in the
2 core of the article after demolding, these slowly diffuse out of the article without
3 detriment to its usability or recyclability.

4 Excellent dimensional stability of the article is achieved as a result of its
5 closed firm external skin. In addition, foamed injection molded articles which have
6 a homogeneous uniform external skin and excellent tactility can be obtained with the
7 process according to the disclosure.

8 The foamed injection molded articles obtained have an excellent surface
9 quality and do not require any further finishing. It is also of advantage that the cavity
10 does not need to be treated with a separating agent.

11 The process according to the disclosure for the pressure-controlled metered
12 addition of physical propellants to an expandable melt can be conducted
13 advantageously with a device comprising a storage means 11, in which the propellant
14 is stored under pressure, a pressure control valve 10 for regulating the propellant
15 pressure and an injection point 5, which is preferably configured as a throttle means,
16 at which the propellant under pressure is added to the melt, wherein the injection point
17 5 includes a controlled closure mechanism, and in the case of critical propellants at
18 least one pressure relief valve 4 is provided which is positioned downstream of the
19 pressure control valve 10.

20 Although the above-described process and the device for the pressure-
21 controlled metered addition of propellants under high pressure can be advantageously
22 used for the production of physically foamed injection molded articles, they are, of
23 course, also suitable for other processes in which propellants are added under high
24 pressure to melts to be expanded.

25 What is claimed is:

Process for the Production of Physically Foamed Injection Moulded Articles

The present invention relates to a process for the production of physically foamed injection moulded articles, in particular injection moulded articles with an internal foam structure and a compact closed-pore external skin of the same material as the base body.

The production of foamed plastics, for example, is achieved with the aid of so-called propellants, which expand a plastic, generally thermally softened plastic mass in the desired manner. In this case, the propellants are either generated in situ via chemical reaction of the components (chemical propellants), or compressed fluids, e.g. N₂, CO₂, are added under pressure to the starting material, in which case a foaming process of the plastic mass caused by the propellant is initiated upon the subsequent drop in pressure of the component mixture to normal pressure.

However, chemical propellants have a series of disadvantages. For instance, for use in foam injection moulding higher temperatures than are actually necessary for softening starting materials may have to be selected in order to reach the ignition point of the propellants, since the temperature at which the reaction of the components generating propellant starts is generally very high. Because of the high temperatures a higher expenditure of energy is necessary during melting of the raw materials. In addition, the cycle or cooling times are increased and a higher cooling power of the cooling plants is necessary. In some circumstances, damage to the raw materials may also occur as a result of the comparatively high temperatures.

Chemical propellants which have not been converted can locate on the surface of the articles obtained and cause yellowing of

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the articles. Allergic skin reactions may also result upon contact with these articles.

Foam articles which have been obtained by means of chemical propellants are not recyclable, or if so only conditionally, since there is the risk that non-ignited propellants can lead to uncontrolled reactions during reuse.

Therefore, physical propellants are preferably used to foam plastics. Physical propellants allow optimum adaptation of the melting temperature to the respectively selected raw material, as a result of which the energy expenditure is reduced, optimum cycle and cooling times are made possible and in addition there is no risk that the raw materials could be detrimentally affected as a result of temperatures which are too high. Moreover, inexpensive gases such as CO_2 , for example, can be used as physical propellants.

Physical propellants do not remain in the finished foam articles, but diffuse out within a comparatively short time. Therefore, these articles are fully recyclable, since there is no need to fear that propellant residues could lead to uncontrolled reactions.

Various processes are known for the production of articles from foamed plastic with a compact closed external skin and a cellular core cohering with the external skin or edge zone, also referred to as integral foam or structural foam.

For example, in the reaction injection moulding process (RIM), two reactive components are mixed together which harden and foam in the cavity of a mould under reaction. Because of the quicker cooling at the wall of the mould, the reaction mass solidifies more quickly there than in the interior of the mould, and as a result the foaming process ceases earlier there than in the mould interior, and a compact sealed external layer is formed.

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As determined by the process, the reactive component mixture must be comparatively liquid in order to guarantee complete filling of the mould before the reaction starts. However, this leads to irregularities on the surface of the formed article as a result of spray over and skin formation, which necessitates expensive finishing for high-grade articles, for which a perfect surface is required.

Moreover, for the RIM process the mould must be treated with a separating agent prior to injection, which on the one hand requires more expenditure in processing and can additionally lead to residues on the finished article which must be removed. The relatively long cycle times are also disadvantageous.

Since foaming in the RIM process is generally conducted chemically, the articles to be obtained are only conditionally recyclable.

Integral foams made of polyurethane to be used as working material primarily in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are preferably produced using the RIM process. However, for this field of application the articles must not only have as perfect a surface as possible, but also have pleasant skin feel (tactility).

It has been shown that articles of polyurethane integral foam have only a conditionally acceptable tactility.

It is also known to produce integral foams from thermoplastic urethane or thermoplastic elastomer by means of conventional injection moulding processes. Both chemical and physical propellants can be used in this case. Contrary to the RIM process, which requires special plants, already existing injection moulding plants without expensive refitting can be used for this.

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The necessary finishing of the articles obtained is only slight.

DE 196 46 665 A1 describes a process for metering physical propellants, wherein a propellant is added at high pressure to the softened plastic material transported in the consumer, e.g. an extruder or an RIM machine, and the amount of propellant is regulated with a pressure control valve, which keeps the pressure difference constant via a rigid throttle means by regulating the pressure difference in dependence on the flow of propellant. The extrusion processes described here are continuous processes in which the propellant is permanently added.

A process for the production of multilayered articles with a foamed core and a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a first propellant-free melt and a second melt containing propellant as well as possibly a third propellant-free melt are firstly prepared and injected one after the other into an appropriate mould, in which case the mould must be maintained at a temperature equal to or higher than the activation temperature of the propellant.

Where physical propellants are used, it is suggested that either the selected temperature of the melt upon leaving the nozzle is so high that, when a mould with constant internal volume is used, the gas formation, and thus the expansion, still occurs below the pressure exerted on the substance in the mould, and when a mould with extendable interior is used, the gas formation, and thus the expansion, occurs by relieving the pressure exerted on the mould interior to expand the mould. There is no mention of the propellant being added directly to the melt flow which flows into the mould, nor of the quantity of propellant apportioned to the melt flow being regulated via the pressure.

An improved process of the aforementioned type is specified in DE 1 948 454, wherein the propellant is injected into the melt

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flow shortly before entry into the mould and the injection period is continued until the mixture quantity required to form the core has been inserted into the mould. Solvents with a boiling point preferably between 20 and 150°C are specified as propellants, which are to prevent premature expansion under a corresponding pressure. There is likewise no mention here of a pressure regulation of the added quantity of propellant to the melt.

The object of the present invention is to provide a process for the production of physically foamed injection moulded articles, with which injection moulded articles with an integral structure, excellent surface characteristics, thus rendering expensive finishing unnecessary, and additionally excellent tactility, can be obtained in a simple manner using conventional injection moulding plants.

The articles produced according to the invention are suitable in particular for fields of application which set high quality requirements for surface structure and for which a pleasant sensory feel is of advantage on skin contact. The automobile industry is given as an example, for which handles, knobs such as gearshift knobs, steering wheel casings etc. of the foamed plastics obtained according to the invention can be used. However, the process according to the invention is in no way restricted to the production of articles for the automobile industry, but is quite generally suitable for the production of any desired foamed injection moulded articles.

For example, mass-produced articles such as closing means for bottle-like containers, e.g. stoppers or corks, may also be advantageously obtained according to this process. Further examples are balls, spheres, fenders, floats etc.

A further field of use is the production of supporting parts, for example, for the aviation or automobile industry, in particular for parts where strength is relevant.

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This object is achieved according to the invention by a process for the production of physically foamed injection moulded articles, wherein firstly in a first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), wherein metering of the physical propellant occurs at least in a pressure regulated manner, and possibly in a third stage a propellant-free further melt portion is charged into the cavity.

The present invention also relates to physically foamed injection moulded articles, the foamed core of which is completely or partially enclosed by a compact closed external skin, which has been produced without the addition of propellants, the core and the external skin being made of the same material.

The present invention additionally relates to a device for the metered addition of propellants under elevated pressure to an expandable melt.

This device can also be advantageously used for the metered addition of compressible propellants.

The propellant-free melt portion firstly fed into the cavity in the first stage forms a compact closed external skin without pores in the finished foamed injection moulded articles.

Any desired fluid which expands upon corresponding pressure relief and foams the melt material in a suitable manner can be used as propellant. Hence, compressible fluids such as gases in liquid or supercritical phase, for example, may be used.

The use of carbon dioxide is recommended because of its ready availability.

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A further preferred propellant is water.

The starting material for the melt is not subject to any special restrictions. Any desired thermoplastic melt material which is suitable for injection moulding and can be foamed may be used.

Examples are thermoplastic materials, but also further thermoplastic melts, such as metallic or ceramic melts, for example. Examples of metallic materials include aluminium, magnesium, zinc, tin or even precious metals.

The process according to the invention leads to weight reduction and strength increase in comparison to the corresponding compact articles.

"Pressure regulated" in the sense of the invention means that in the course of the process the pressure exerted on the propellant is varied for metered addition of the propellant. In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve.

Therefore, according to the invention the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure.

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present invention is explained in more detail below with reference to the figures on the basis of a preferred

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Figures 1a-1d show the individual stages of the process according to the invention for the production of physically foamed injection moulded articles;

Figure 3 is a graph showing the pressure curve during execution of the process;

As Figure 1a shows, the cavity 1 of any injection moulding plant is partially initially filled in a first stage firstly with compact propellant-free melt 6. In this case, the feed pipe 3 for a compressed propellant is closed, for example, by a valve 4 such as a pressure relief valve (overflow valve).

As a general rule the propellant is still liquid and not gaseous at the injection point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower sense.

The mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes the cavity 1 to fill completely, in which

case the propellant-free melt portion 6 which was used for the initial filling comes to rest in the region of the cavity walls and forms the external skin or edge zone of the injection moulded article to be formed.

The cavity 1 can be ready filled as desired and required up to the maximum filling quantity with melt mixed with propellant or, as shown in Figure 1d, propellant-free melt can again be fed to the cavity in a third stage. In this case a foamed article is obtained which has a compact firm external skin right around which is formed by propellant-free melt.

After foaming and hardening, the finished injection moulded article, e.g. made of integral foam, is removed from the cavity and the cavity is immediately available again for the next charge.

As shown in Figure 1d, injection moulded articles, which have a cellular foamed internal core and a compact firm closed external skin, are obtained with the process according to the invention.

Contrary to the known foaming processes, such as those described above, in which the cavity is filled completely with a melt/propellant mixture, according to the invention an initial filling with propellant-free melt occurs firstly, as a result of which the formation of a uniform closed compact external skin is effected and articles with excellent surface characteristics can be obtained.

It is essential for execution of the process to prevent premature expansion of the propellant held under pressure. This can be achieved by appropriate insulation of the device and/or maintaining a suitable pressure level.

The metered addition of the propellant is conducted in a time- and pressure-controlled manner for the process according to

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the invention. Control can be carried out via a device which is also the subject of the invention.

As shown in Figure 2, the propellant stored under pressure in a storage means 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a multi-way valve such as a 3/3- or 2/3-way proportional valve, and should advantageously have a very quick reaction time and precise regulation.

During the propellant injection phase, i.e. the phase in which the propellant is added to the melt, in the case of critical propellants, the compressed propellant passes via a pressure relief valve 4 to the injection point 5 and there is added to the melt.

In this case, the dimensions of the pipes, connection pieces and also the parts of the technical control system of the process are such that no premature expansion in volume of the propellant under pressure is possible.

In the case of a sudden increase in volume the aggregate state of the agent can change, i.e. the agent changes into a gas, in which case vaporisation cold is generated, which would in turn block the pipes as a result of "icing".

An increase in temperature on the way to the injection point 5 would also lead to a change in the aggregate state. For prevention, insulation of the heat-carrying elements is recommended.

In order to prevent premature expansion, all feed pipes should be as short as possible. Consequently, the pressure control valve 10 is preferably constructed to be as close as possible to the injection point 5. An improvement to the control characteristics of the control valve is also achieved as a result of the thus shortened feed pipe to the injection point 5.

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If critical propellants are used, a pressure relief valve or overflow valve 4 is provided before the injection point 5, this valve ensuring that the pressure in the device does not drop below a specific value, preferably p_{crit} at the given temperature, at which the transformation of the propellant into gas would take place. If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar should be maintained at room temperature in order to keep the carbon dioxide in the device upstream in liquid state.

The pressure relief valve 4 ensures that the propellant remains in compressed state even during outage times of the machine, e.g. in the intermediate cycle times before and after or between the propellant injection phases. A full release of pressure only occurs when the machine or control system is switched off. Several pressure relief valves with "falling" pressure values may also be provided so that a pressure gradient is formed in front of the injection point 5 in the feed pipe section between the pressure control valve 10 and the pressure relief valve 4.

The graph in Figure 3 schematically shows the pressure curve for executing the process according to the invention using the example of compressible propellants.

Outside of the propellant injection phase, as in the intermediate cycle times, it is sufficient to keep the device at a selected pressure, at which the propellant respectively used remains in compressed, preferably liquid, state (section 20).

During the propellant injection phase (section 22), an elevated pressure is introduced in the feed pipes through the pressure control valve 10 so that the opening pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section 3 up to the injection point 5 is quickly filled with liquid medium.

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In this case, the pressure increase is proportional to the desired quantity of propellant to be fed to the melt. After time t expires, as soon as the desired quantity of propellant has been added to the melt, the pressure is reduced again to the starting pressure (section 24).

In Figure 3, sections 21 and 23 show the pressure build up or reduction phase.

The injection point 5 is preferably configured as a throttle means, e.g. as a defined gap in an injector, a sintered metal injector or a needle valve. A controlled closure mechanism is also particularly recommended for this. The quick pressure increase and the resistance through the injector prevent the propellant from transforming into gas, while the agent flows on from the pressure control valve 10.

The above measures ensure that the transformation of the agent into gas only occurs upon exit from the injector and when in contact with the hot melt, and that the inflowing melt is foamed.

After the propellant injection phase has ended, i.e. after the desired quantity of propellant has been added to the melt, the pressure in the feed pipe to the injection point 5 is reduced so that no propellant can flow on. However, in the pipe up to the pressure relief valve 4 the starting pressure remains in order to keep the agent in compressed or liquid state for the next cycle. A virtually pressure-free and thus gaseous state prevails only in the short feed pipe section from the pressure relief valve 4 to the injection point 5 until the next cycle. It goes without saying that this part of the plant may also be kept under pressure if required by the provision of a suitable closure mechanism which opens again at the beginning of the propellant injection phase as a result of the increasing pressure level.

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The pressure control via the pressure control valve can occur automatically by providing pressure measurement points 12, 13, for example, in front of and behind the pressure control valve.

If carbon dioxide is used as propellant, for example, the plant is preferably held at an operating pressure of at least 60 bar at room temperature, so that the CO₂ also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure of about 200 bar, for example, is built up (section 21) in order to assure an adequate flow of propellant to the melt. After the propellant injection phase 22 has ended, the pressure is reduced again to the desired operating pressure.

The injection point 5 is preferably located in the feeder pipe 3 close to the spray point x. According to a further embodiment, as is shown in Figure 4, the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity.

In addition, the build up of a counterpressure can be provided in the cavity 1, such as is also used in conventional injection moulding processes in the so-called gas counterpressure process.

Very short cycle times can be obtained with the process according to the invention. Hence, the process according to the invention is also very well suited to the production of mass-produced articles. The short cycle times are supported by the vaporisation cold resulting upon the transformation of the propellant into gas, and this causes a reduction in the cooling time, and thus also the cycle time.

Should there still be propellant residues present in the pore structure in the core of the article after demoulding, these

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slowly diffuse out of the article without detriment to its usability or recyclability.

Excellent dimensional stability of the article is achieved as a result of its closed firm external skin. In addition, foamed injection moulded articles which have a homogeneous uniform external skin and excellent tactility can be obtained with the process according to the invention.

The foamed injection moulded articles obtained have an excellent surface quality and do not require any further finishing. It is also of advantage that the cavity does not need to be treated with a separating agent.

The process according to the invention for the pressure-controlled metered addition of physical propellants to an expandable melt can be conducted advantageously with a device comprising a storage means 11, in which the propellant is stored under pressure, a pressure control valve 10 for regulating the propellant pressure and an injection point 5, which is preferably configured as a throttle means, at which the propellant under pressure is added to the melt, wherein in the case of critical propellants at least one pressure relief valve 4 is provided which is positioned downstream of the pressure control valve 10.

Although the above-described process and the device for the pressure-controlled metered addition of propellants under high pressure can be advantageously used for the production of physically foamed injection moulded articles, they are, of course, also suitable for other processes in which propellants are added under high pressure to melts to be expanded.

List of Reference Numbers

1	cavity
2	melt feed
3	propellant feed pipe
4	pressure relief valve
5	injection point
6	propellant-free melt
7	melt with added propellant
8	injection of plastic material
9	mould comprising two halves
10	pressure control valve
11	propellant storage means
x	spray point
Section 20	pressure during the intermediate cycle times
Section 21	pressure build up phase
Section 22	propellant injection phase
Section 23	pressure reduction phase

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Claims:

1. Process for the production of physically foamed injection moulded articles, characterised in that in a first stage a propellant-free first melt portion (6) is fed into a cavity (1) (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), wherein the dosage of the physical propellant occurs at least in a pressure regulated manner, and possibly in a third stage a propellant-free further melt portion is charged into the cavity (1).
2. Process according to Claim 1, characterised in that the propellant is a compressible fluid.
3. Process according to Claim 1 or 2, characterised in that the propellant is kept under pressure in the intermediate cycle times before and after the propellant injection phase, or is present in a compressed state.
4. Process according to Claim 3, characterised in that in the intermediate cycle times the propellant is held a pressure of at least p (crit) of the propellant at the given temperature.
5. Process according to one of the preceding claims, characterised in that the pressure exerted on the propellant is controlled via a pressure control valve (10).
6. Process according to Claim 5, characterised in that the pressure control valve (10) is a multi-way valve.
7. Process according to Claim 6, characterised in that a 3/3-way proportional valve or a 2/3-way proportional valve is used as multi-way valve.

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8. Process according to one of the preceding claims, characterised in that the pressure control in the case of critical propellants additionally occurs via at least one pressure relief valve (4) which is connected downstream of the pressure control valve (10).
9. Process according to Claim 8, characterised in that the holding pressure of at least one of the pressure relief valves (4) is equal to or higher than the pressure at which a critical propellant is held in the intermediate cycle times.
10. Process according to one of the preceding claims, characterised in that the pressure preset by the pressure control valve (10) is regulated via one or more pressure relief valves (4) to the injection pressure at which the propellant is added to the melt via an injection point (5).
11. Process according to one of the preceding claims, characterised in that the injection point (5) is configured as a throttle means.
12. Process according to Claim 11, characterised in that the injection point (5) is in the form of a defined gap in an injector or of an injector with a sinter metal.
13. Process according to one of Claims 11 or 12, characterised in that the injection point (5) is configured as a controlled closure mechanism.
14. Process according to Claim 1 or one of the preceding Claims 3 to 13, characterised in that water is used as propellant.
15. Process according to one of the preceding Claims 1 to 13, characterised in that a gas or gas mixture is used as propellant.

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16. Process according to Claims 15, characterised in that carbon dioxide is used as propellant.
17. Process according to Claims 16, characterised in that the carbon dioxide is held in the intermediate cycle times at a pressure of at least 60 bar (= p (crit) CO₂ at room temperature).
18. Process according to one of the preceding claims, characterised in that for the propellant injection phase the propellant is brought to a pressure of over 60 bar via the pressure control valve (10).
19. Process according to one of the preceding claims, characterised in that a counterpressure is generated in the cavity (1).
20. Physically foamed injection moulded article with an external skin and a foamed core of the same material, characterised in that the foamed core is enclosed completely or partially by the external skin and the external skin is configured as a compact closed envelope without the addition of propellant.
21. Physically foamed injection moulded article according to Claim 20, characterised in that the injection moulded article is a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a fender or a float.
22. Physically foamed injection moulded article according to Claim 20, characterised in that the injection moulded article is a closing means for bottle-like containers such as a stopper or a cork.
23. Use of an injection moulded article according to one of Claims 20 or 21 in the automobile industry.

- # Orangeburg

1 AS AMENDED

Process for the Production of Physically Foamed Injection Moulded Articles

The present invention relates to a process for the production of physically foamed injection moulded articles, in particular injection moulded articles with an internal foam structure and a compact closed-pore external skin of the same material as the base body.

The production of foamed plastics, for example, is achieved with the aid of so-called propellants, which expand a plastic, generally thermally softened plastic mass in the desired manner. In this case, the propellants are either generated in situ via chemical reaction of the components (chemical propellants), or compressed fluids, e.g. N_2 , CO_2 , are added under pressure to the starting material, in which case a foaming process of the plastic mass caused by the propellant is initiated upon the subsequent drop in pressure of the component mixture to normal pressure.

However, chemical propellants have a series of disadvantages. For instance, for use in foam injection moulding higher temperatures than are actually necessary for softening starting materials may have to be selected in order to reach the ignition point of the propellants, since the temperature at which the reaction of the components generating propellant starts is generally very high. Because of the high temperatures a higher expenditure of energy is necessary during melting of the raw materials. In addition, the cycle or cooling times are increased and a higher cooling power of the cooling plants is necessary. In some circumstances, damage to the raw materials may also occur as a result of the comparatively high temperatures.

Chemical propellants which have not been converted can locate on the surface of the articles obtained and cause yellowing of the articles. Allergic skin reactions may also result upon contact with these articles.

Foam articles which have been obtained by means of chemical propellants are not recyclable, or if so only conditionally, since there is the risk that non-ignited propellants can lead to uncontrolled reactions during reuse.

Therefore, physical propellants are preferably used to foam plastics. Physical propellants allow optimum adaptation of the melting temperature to the respectively selected raw material, as a result of which the energy expenditure is reduced, optimum cycle and cooling times are made possible and in addition there is no risk that the raw materials could be detrimentally affected as a result of temperatures which are too high. Moreover, inexpensive gases such as CO_2 , for example, can be used as physical propellants.

Physical propellants do not remain in the finished foam articles, but diffuse out within a comparatively short time. Therefore, these articles are fully recyclable, since there is no need to fear that propellant residues could lead to uncontrolled reactions.

Various processes are known for the production of articles from foamed plastic with a compact closed external skin and a cellular core cohering with the external skin or edge zone, also referred to as integral foam or structural foam.

For example, in the reaction injection moulding process (RIM), two reactive components are mixed together which harden and foam in the cavity of a mould under reaction. Because of the quicker cooling at the wall of the mould, the reaction mass solidifies more quickly there than in the interior of the mould, and as a result the foaming process ceases earlier there than in the mould interior, and a compact sealed external layer is formed.

As determined by the process, the reactive component mixture must be comparatively liquid in order to guarantee complete filling of the mould before the reaction starts. However, this leads to irregularities on the surface of the formed article as a result of spray over and skin formation, which necessitates expensive finishing for high-grade articles, for which a perfect surface is required.

Moreover, for the RIM process the mould must be treated with a separating agent prior to injection, which on the one hand requires more expenditure in processing and can additionally lead to residues on the finished article which must be removed. The relatively long cycle times are also disadvantageous.

Since foaming in the RIM process is generally conducted chemically, the articles to be obtained are only conditionally recyclable.

Integral foams made of polyurethane to be used as working material primarily in the automobile industry, e.g. for steering wheel casings or gearshift knobs etc., are preferably produced using the RIM process. However, for this field of application the articles must not only have as perfect a surface as possible, but also have pleasant skin feel (tactility).

It has been shown that articles of polyurethane integral foam have only a conditionally acceptable tactility.

5 It is also known to produce integral foams from thermoplastic urethane or thermoplastic elastomer by means of conventional injection moulding processes. Both chemical and physical propellants can be used in this case. Contrary to the RIM process, which requires special plants, already existing injection moulding plants without expensive refitting can be used for this.

The necessary finishing of the articles obtained is only slight.

DE 196 46 665 A1 describes a process for metering physical propellants, wherein a propellant is added at high pressure to the softened plastic material transported in the consumer, e.g. an extruder or an RIM machine, and the amount of propellant is regulated with a pressure control valve, which keeps the pressure difference constant via a rigid throttle means by regulating the pressure difference in dependence on the flow of propellant. The extrusion processes described here are continuous processes in which the propellant is permanently added.

25 A process for the production of multilayered articles with a foamed core and a non-expanded thermoplastic external skin is known from DE 1 778 457, wherein a first propellant-free melt and a second melt containing propellant as well as possibly a third propellant-free melt are firstly prepared and injected one after the other into an appropriate mould, in which case the mould must be maintained at a temperature equal to or higher than the activation temperature of the propellant.

Where physical propellants are used, it is suggested that either the selected temperature of the melt upon leaving the nozzle is so high that, when a mould with constant internal volume is used, the gas formation, and thus the expansion, still occurs below the pressure exerted on the substance in the mould, and when a mould with extendable interior is used, the gas formation, and thus the expansion, occurs by relieving the pressure exerted on the mould interior to expand the mould. There is no mention of the propellant being added directly to the melt flow which flows into the mould, nor of the quantity of propellant apportioned to the melt flow being regulated via the pressure.

An improved process of the aforementioned type is specified in DE 1 948 454, wherein the propellant is injected into the melt flow shortly before entry into the mould and the injection period is continued until the mixture quantity required to form the core has been inserted into the mould. Solvents with a boiling point preferably between 20 and 150°C are specified as propellants, which are to prevent premature expansion under a corresponding pressure. There is likewise no mention here of a pressure regulation of the added quantity of propellant to the melt.

A process for the production of injection moulded articles with foamed core is described in the US patent 4,548,776, according to which gaseous or gas-generated chemical propellant is already added to the melt in the extruder, is thoroughly mixed with this and the already foamed melt is then injected into the mould.

In this case, the addition of propellant occurs via a porous insert at the injection point, a supply valve being provided in the feed

pipe. This supply valve can be connected to an automatic control device, via which the pressure of the propellant to be fed is adjusted.

5 The object of the present invention is to provide a process for the production of physically foamed injection moulded articles, with which injection moulded articles with an integral structure, excellent surface characteristics, thus rendering expensive finishing unnecessary, and additionally excellent tactility, can be
10 obtained in a simple manner using conventional injection moulding plants.

15 The articles produced according to the invention are suitable in particular for fields of application which set high quality requirements for surface structure and for which a pleasant sensory feel is of advantage on skin contact. The automobile industry is given as an example, for which handles, knobs such as gearshift knobs, steering wheel casings etc. of the foamed plastics obtained
20 according to the invention can be used. However, the process according to the invention is in no way restricted to the production of articles for the automobile industry, but is quite generally suitable for the production of any desired foamed injection moulded articles.

25 For example, mass-produced articles such as closing means for bottle-like containers, e.g. stoppers or corks, may also be advantageously obtained according to this process. Further examples are balls, spheres, fenders, floats etc.

A further field of use is the production of supporting parts, for example, for the aviation or automobile industry, in particular for parts where strength is relevant.

5 This object is achieved according to the invention by a process for the production of physically foamed injection moulded articles, wherein firstly in a first stage a propellant-free first melt portion is fed into a cavity (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), wherein metering of the physical propellant occurs at least in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity, and possibly in a third stage a propellant-free further melt portion is charged into the cavity.

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20 This process also permits the formation of physically foamed injection moulded articles, the foamed core of which is completely or partially enclosed by a compact closed external skin, which has been produced without the addition of propellants, the core and the external skin being made of the same material.

25 The present invention additionally relates to a device for the metered addition of propellants under elevated pressure to an expandable melt.

30 This device can also be advantageously used for the metered addition of compressible propellants.

The propellant-free melt portion firstly fed into the cavity in the first stage forms a compact closed external skin without pores in the finished foamed injection moulded articles.

5 Any desired fluid which expands upon corresponding pressure relief and foams the melt material in a suitable manner can be used as propellant. Hence, compressible fluids such as gases in liquid or supercritical phase, for example, may be used.

10 The use of carbon dioxide is recommended because of its ready availability.

A further preferred propellant is water.

15 The starting material for the melt is not subject to any special restrictions. Any desired thermoplastic melt material which is suitable for injection moulding and can be foamed may be used.

20 Examples are thermoplastic materials, but also further thermoplastic melts, such as metallic or ceramic melts, for example. Examples of metallic materials include aluminium, magnesium, zinc, tin or even precious metals.

25 The process according to the invention leads to weight reduction and strength increase in comparison to the corresponding compact articles.

30 "Pressure regulated" in the sense of the invention means that in the course of the process the pressure exerted on the propellant is varied for metered addition of the propellant.

In this case the pressure exerted on the propellant during the propellant injection phase is greater than the pressure exerted on the propellant in the phases between or before or after metered addition. This means in the case of critical or compressible propellants, for example, that the pressure exerted in the intermediate cycle times is lower than the holding pressure of a pressure relief valve or overflow valve.

Therefore, according to the invention the required proportion of propellant is added to a melt to be foamed at a defined time over a defined period of time under a defined pressure.

The magnitude of the pressure exerted on the propellant during the metered addition is determined in particular in dependence on the required quantity of propellant, the type of article to be produced as well as the selected process parameters.

The present invention is explained in more detail below with reference to the figures on the basis of a preferred embodiment by the example of the addition of a compressible fluid. It goes without saying that the following explanation may also be applied in principle to non-compressible fluids such as water, for example.

Figures 1a-1d show the individual stages of the process according to the invention for the production of physically foamed injection moulded articles;

Figure 2 schematically shows a device for executing the process according to the invention;

Figure 3 is a graph showing the pressure curve during execution of the process;

Figure 4 shows a variant of Figure 1 with direct introduction of the propellant into the cavity.

As Figure 1a shows, the cavity 1 of any injection moulding plant is partially initially filled in a first stage firstly with compact propellant-free melt 6. In this case, the feed pipe 3 for a compressed propellant is closed, for example, by a valve 4 such as a pressure relief valve (overflow valve).

After the cavity 1 has been filled with a desired quantity of propellant-free melt 6, the feed pipe 3 for the propellant is opened and the propellant is injected in compressed, preferably liquid, state via the injection point 5. Through contact with the hot melt, the liquid propellant turns to gas and expands under the lower pressure in the cavity.

As a general rule the propellant is still liquid and not gaseous at the injection point 5 itself, and therefore one cannot talk of a "gasification point" in a narrower sense.

The mixture 7 of gaseous propellant and melt flows into the cavity 1 and causes the cavity 1 to fill completely, in which case the propellant-free melt portion 6 which was used for the initial filling comes to rest in the region of the cavity walls and forms the external skin or edge zone of the injection moulded article to be formed.

The cavity 1 can be ready filled as desired and required up to the maximum filling quantity with melt mixed with propellant or, as shown in Figure 1d, propellant-free melt can again be fed to the cavity in a third stage. In this case a foamed article is obtained which has a compact firm external skin right around which is formed by propellant-free melt.

After foaming and hardening, the finished injection moulded article, e.g. made of integral foam, is removed from the cavity and the cavity is immediately available again for the next charge.

As shown in Figure 1d, injection moulded articles, which have a cellular foamed internal core and a compact firm closed external skin, are obtained with the process according to the invention. Contrary to the known foaming processes, such as those described above, in which the cavity is filled completely with a melt/propellant mixture, according to the invention an initial filling with propellant-free melt occurs firstly, as a result of which the formation of a uniform closed compact external skin is effected and articles with excellent surface characteristics can be obtained.

It is essential for execution of the process to prevent premature expansion of the propellant held under pressure. This can be achieved by appropriate insulation of the device and/or maintaining a suitable pressure level.

The metered addition of the propellant is conducted in a time- and pressure-controlled manner for the process according to the invention. Control can be carried out via a device which is also the subject of the invention.

As shown in Figure 2, the propellant stored under pressure in a storage means 11, e.g. a pressure cylinder etc., is fed to a pressure control valve 10, which can be a multi-way valve such as a 3/3- or 2/3-way proportional valve, and should advantageously have a very quick reaction time and precise regulation.

During the propellant injection phase, i.e. the phase in which the propellant is added to the melt, in the case of critical propellants, the compressed propellant passes via a pressure relief valve 4 to the injection point 5 and there is added to the melt.

In this case, the dimensions of the pipes, connection pieces and also the parts of the technical control system of the process are such that no premature expansion in volume of the propellant under pressure is possible.

In the case of a sudden increase in volume the aggregate state of the agent can change, i.e. the agent changes into a gas, in which case vaporisation cold is generated, which would in turn block the pipes as a result of "icing".

An increase in temperature on the way to the injection point 5 would also lead to a change in the aggregate state. For prevention, insulation of the heat-carrying elements is recommended.

In order to prevent premature expansion, all feed pipes should be as short as possible. Consequently, the pressure control valve 10 is preferably constructed to be as close as possible to the injection point 5. An improvement to the control characteristics of

the control valve is also achieved as a result of the thus shortened feed pipe to the injection point 5.

5 If critical propellants are used, a pressure relief valve or overflow valve 4 is provided before the injection point 5, this valve ensuring that the pressure in the device does not drop below a specific value, preferably $p(\text{crit})$ at the given temperature, at which the transformation of the propellant into gas would take place. If, for example, carbon dioxide is used as propellant, a pressure of at least 60 bar should be maintained at room temperature in order to keep the carbon dioxide in the device upstream in liquid state.

10 The pressure relief valve 4 ensures that the propellant remains in compressed state even during outage times of the machine, e.g. in the intermediate cycle times before and after or between the propellant injection phases. A full release of pressure only occurs when the machine or control system is switched off. Several pressure relief valves with "falling" pressure values may also be provided so that a pressure gradient is formed in front of the injection point 5 in the feed pipe section between the pressure control valve 10 and the pressure relief valve 4.

15 The graph in Figure 3 schematically shows the pressure curve for executing the process according to the invention using the example of compressible propellants.

20 Outside of the propellant injection phase, as in the intermediate cycle times, it is sufficient to keep the device at a selected pressure, at which the propellant respectively used remains in compressed, preferably liquid, state (section 20).

During the propellant injection phase (section 22), an elevated pressure is introduced in the feed pipes through the pressure control valve 10 so that the opening pressure (holding pressure) of the relief valve 4 is exceeded and the feed pipe section 3 up to the injection point 5 is quickly filled with liquid medium.

In this case, the pressure increase is proportional to the desired quantity of propellant to be fed to the melt. After time t expires, as soon as the desired quantity of propellant has been added to the melt, the pressure is reduced again to the starting pressure (section 24).

In Figure 3, sections 21 and 23 show the pressure build up or reduction phase.

The injection point 5 is preferably configured as a throttle means, e.g. as a defined gap in an injector, a sintered metal injector or a needle valve. According to the invention, a controlled closure mechanism is located at the injection point. The quick pressure increase and the resistance through the injector prevent the propellant from transforming into gas, while the agent flows on from the pressure control valve 10.

The above measures ensure that the transformation of the agent into gas only occurs upon exit from the injector and when in contact with the hot melt, and that the inflowing melt is foamed.

The controlled closure mechanism can be omitted if a pressure relief valve is provided.

After the propellant injection phase has ended, i.e. after the desired quantity of propellant has been added to the melt, the pressure in the feed pipe to the injection point 5 is reduced so that no propellant can flow on. However, in the pipe up to the pressure relief valve 4 the starting pressure remains in order to keep the agent in compressed or liquid state for the next cycle. A virtually pressure-free and thus gaseous state prevails only in the short feed pipe section from the pressure relief valve 4 to the injection point 5 until the next cycle.

It goes without saying that this part of the plant may also be kept under pressure if required by the provision of a suitable closure mechanism which opens again at the beginning of the propellant injection phase as a result of the increasing pressure level.

The pressure control via the pressure control valve can occur automatically by providing pressure measurement points 12, 13, for example, in front of and behind the pressure control valve.

If carbon dioxide is used as propellant, for example, the plant is preferably held at an operating pressure of at least 60 bar at room temperature, so that the CO_2 also remains in compressed state during the periods between the propellant injection phases. At the beginning of the propellant injection phase, a desired working pressure of about 200 bar, for example, is built up (section 21) in order to assure an adequate flow of propellant to the melt. After the propellant injection phase 22 has ended, the pressure is reduced again to the desired operating pressure.

5 The injection point 5 is preferably located in the feeder pipe 3 close to the spray point x. According to a further embodiment, as is shown in Figure 4, the propellant can be added directly to the melt in the cavity. In this case, the injection point 5 is located directly at the cavity.

In addition, the build up of a counterpressure can be provided in the cavity 1, such as is also used in conventional injection moulding processes in the so-called gas counterpressure process.

10 Very short cycle times can be obtained with the process according to the invention. Hence, the process according to the invention is also very well suited to the production of mass-produced articles. The short cycle times are supported by the vaporisation cold resulting upon the transformation of the propellant into gas, and this causes a reduction in the cooling time, and thus also the cycle time.

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20 Should there still be propellant residues present in the pore structure in the core of the article after demoulding, these slowly diffuse out of the article without detriment to its usability or recyclability.

25 Excellent dimensional stability of the article is achieved as a result of its closed firm external skin. In addition, foamed injection moulded articles which have a homogeneous uniform external skin and excellent tactility can be obtained with the process according to the invention.

30 The foamed injection moulded articles obtained have an excellent surface quality and do not require any further finishing. It is

also of advantage that the cavity does not need to be treated with a separating agent.

5 The process according to the invention for the pressure-controlled metered addition of physical propellants to an expandable melt can be conducted advantageously with a device comprising a storage means 11, in which the propellant is stored under pressure, a pressure control valve 10 for regulating the propellant pressure and an injection point 5, which is preferably configured as a throttle means, at which the propellant under pressure is added to the melt, wherein the injection point 5 includes a controlled closure mechanism, and in the case of critical propellants at least one pressure relief valve 4 is provided which is positioned downstream of the pressure control valve 10.

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20 Although the above-described process and the device for the pressure-controlled metered addition of propellants under high pressure can be advantageously used for the production of physically foamed injection moulded articles, they are, of course, also suitable for other processes in which propellants are added under high pressure to melts to be expanded.

List of Reference Numbers

	1	cavity
5	2	melt feed
	3	propellant feed pipe
	4	pressure relief valve
	5	injection point
	6	propellant-free melt
10	7	melt with added propellant
	8	injection of plastic material
	9	mould comprising two halves
	10	pressure control valve
	11	propellant storage means
15	x	spray point
	Section 20	pressure during the intermediate cycle times
20	Section 21	pressure build up phase
	Section 22	propellant injection phase
	Section 23	pressure reduction phase

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Claims:

1. Process for the production of physically foamed injection moulded articles, wherein in a first stage a propellant-free first melt portion (6) is fed into a cavity (1) (initial filling), in a second stage a physical propellant is added at elevated pressure to the following melt portion (propellant injection phase), and possibly in a third stage a propellant-free further melt portion is charged into the cavity (1), the production of the injection moulded articles occurring in the cavity, characterised in that metering of the physical propellant in the second stage occurs in a pressure regulated manner, wherein the pressure which is exerted on the propellant during the propellant injection phase is greater than the pressure which is exerted on the propellant in the phases between or before or after metered addition, and the expansion of the propellant occurs in the cavity (1).
2. Process according to Claim 1, characterised in that the propellant is a compressible fluid.
3. Process according to Claim 1 or 2, characterised in that the propellant is kept under pressure in the intermediate cycle times before and after the propellant injection phase, or is present in a compressed state.
4. Process according to Claim 3, characterised in that in the intermediate cycle times the propellant is held a pressure of at least p_{crit} of the propellant at the given temperature.

5. Process according to one of the preceding claims, characterised in that the pressure exerted on the propellant is controlled via a pressure control valve (10).
- 5 6. Process according to Claim 5, characterised in that the pressure control valve (10) is a multi-way valve.
7. Process according to Claim 6, characterised in that a 3/3-way proportional valve or a 2/3-way proportional valve is used as multi-way valve.
- 10 8. Process according to one of the preceding claims, characterised in that the pressure control in the case of critical propellants additionally occurs via at least one pressure relief valve (4) which is connected downstream of the pressure control valve (10).
- 15 9. Process according to Claim 8, characterised in that the holding pressure of at least one of the pressure relief valves (4) is equal to or higher than the pressure at which a critical propellant is held in the intermediate cycle times.
- 20 10. Process according to one of the preceding claims, characterised in that the pressure preset by the pressure control valve (10) is regulated via one or more pressure relief valves (4) to the injection pressure at which the propellant is added to the melt via an injection point (5).
- 25 11. Process according to one of the preceding claims, characterised in that the injection point (5) is configured as a throttle means.
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12. Process according to Claim 11, characterised in that the injection point (5) is in the form of a defined gap in an injector or of an injector with a sinter metal.
- 5 13. Process according to one of Claims 11 or 12, characterised in that the injection point (5) is configured as a controlled closure mechanism.
- 10 14. Process according to Claim 1 or one of the preceding Claims 3 to 13, characterised in that water is used as propellant.
- 15 15. Process according to one of the preceding Claims 1 to 13, characterised in that a gas or gas mixture is used as propellant.
- 20 16. Process according to Claims 15, characterised in that carbon dioxide is used as propellant.
- 25 17. Process according to Claims 16, characterised in that the carbon dioxide is held in the intermediate cycle times at a pressure of at least 60 bar (= p (crit) CO₂ at room temperature).
- 30 18. Process according to one of the preceding claims, characterised in that for the propellant injection phase the propellant is brought to a pressure of over 60 bar via the pressure control valve (10).
19. Process according to one of the preceding claims, characterised in that a counterpressure is generated in the cavity (1).
20. Process according to one of the preceding claims,

characterised in that the physically foamed injection moulded article is selected from a handle, a knob, a gearshift knob, a steering wheel casing, a ball, a sphere, a fender, a float and a closing means for bottle-like containers.

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21. Device for the metered addition of physical propellants to a foamable melt, wherein the device comprises a storage means (11), in which the propellant is stored under pressure, a pressure control valve (10) for regulating the propellant pressure, and an injection point (5), which is configured as a throttle means, at which the propellant under pressure is fed to the melt, characterised in that a controlled closure mechanism is provided at the injection point (5).

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22. Device for the metered addition of physical propellants according to Claim 21, characterised in that instead of the controlled closure mechanism or in addition to the controlled closure mechanism, at least one pressure relief valve (4) is provided.

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FIG. 1a

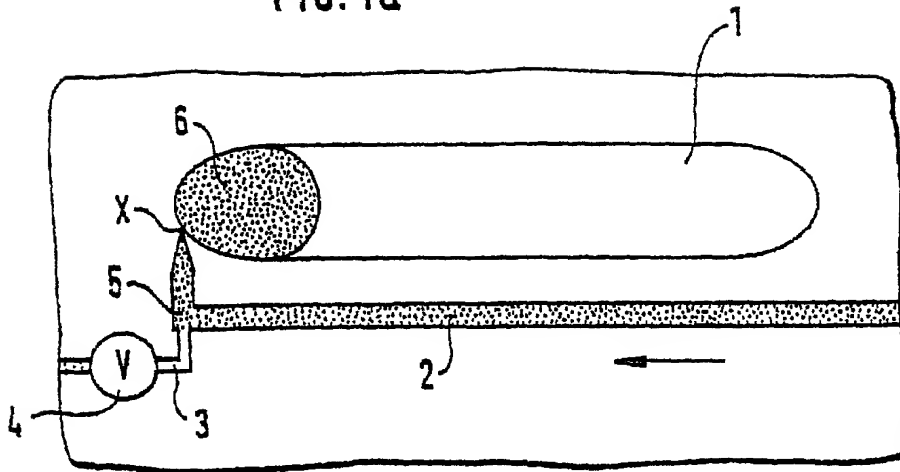


FIG. 1a

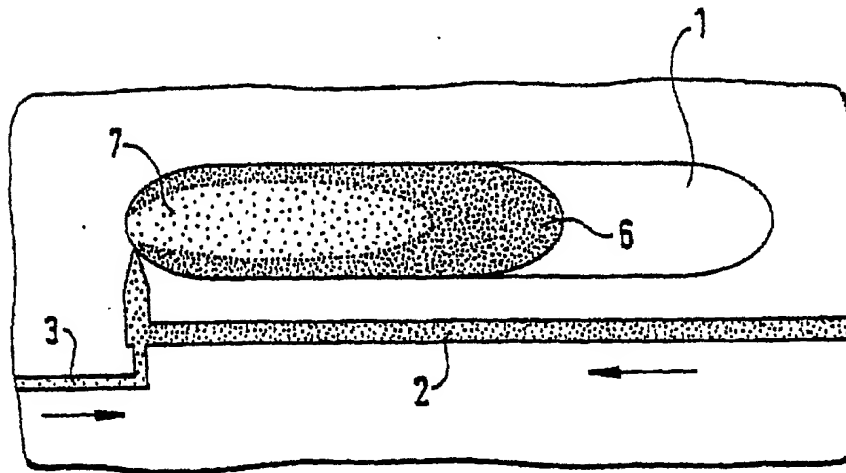


FIG. 1b

FIG. 1b

09/936756, 09/1701

FIG. 1c

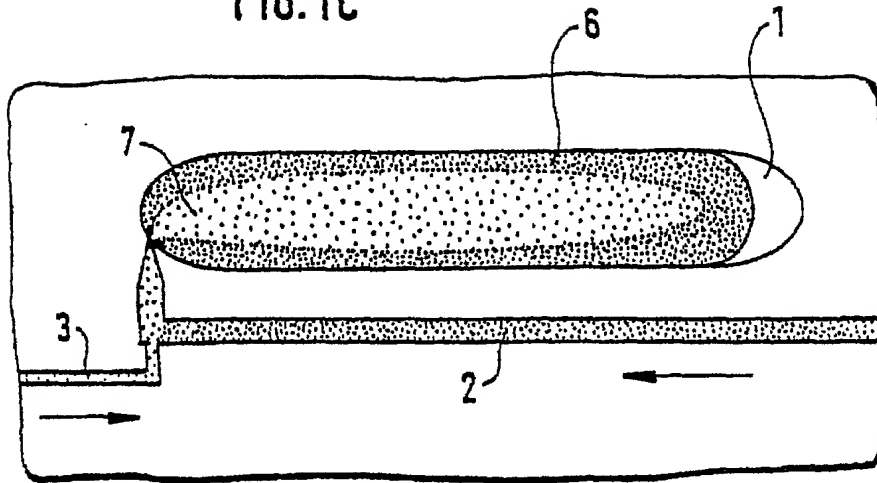


FIG. 1c

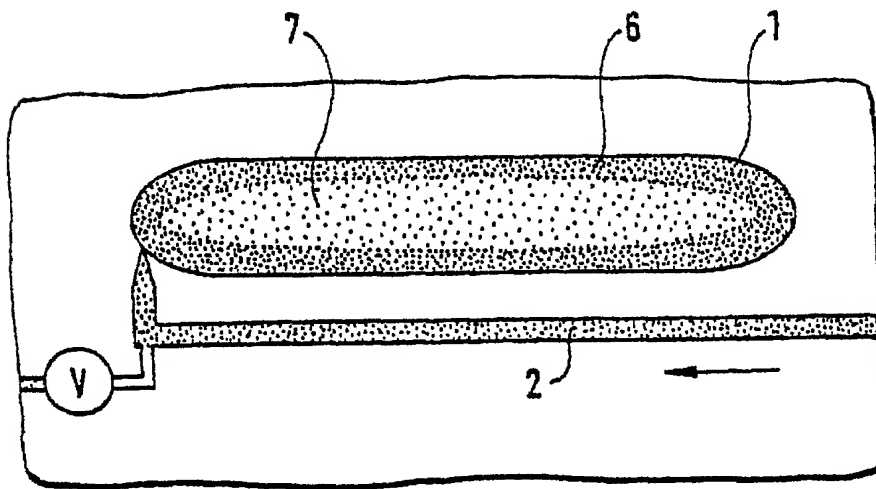


FIG. 1d

FIG. 1d

FIG. 2

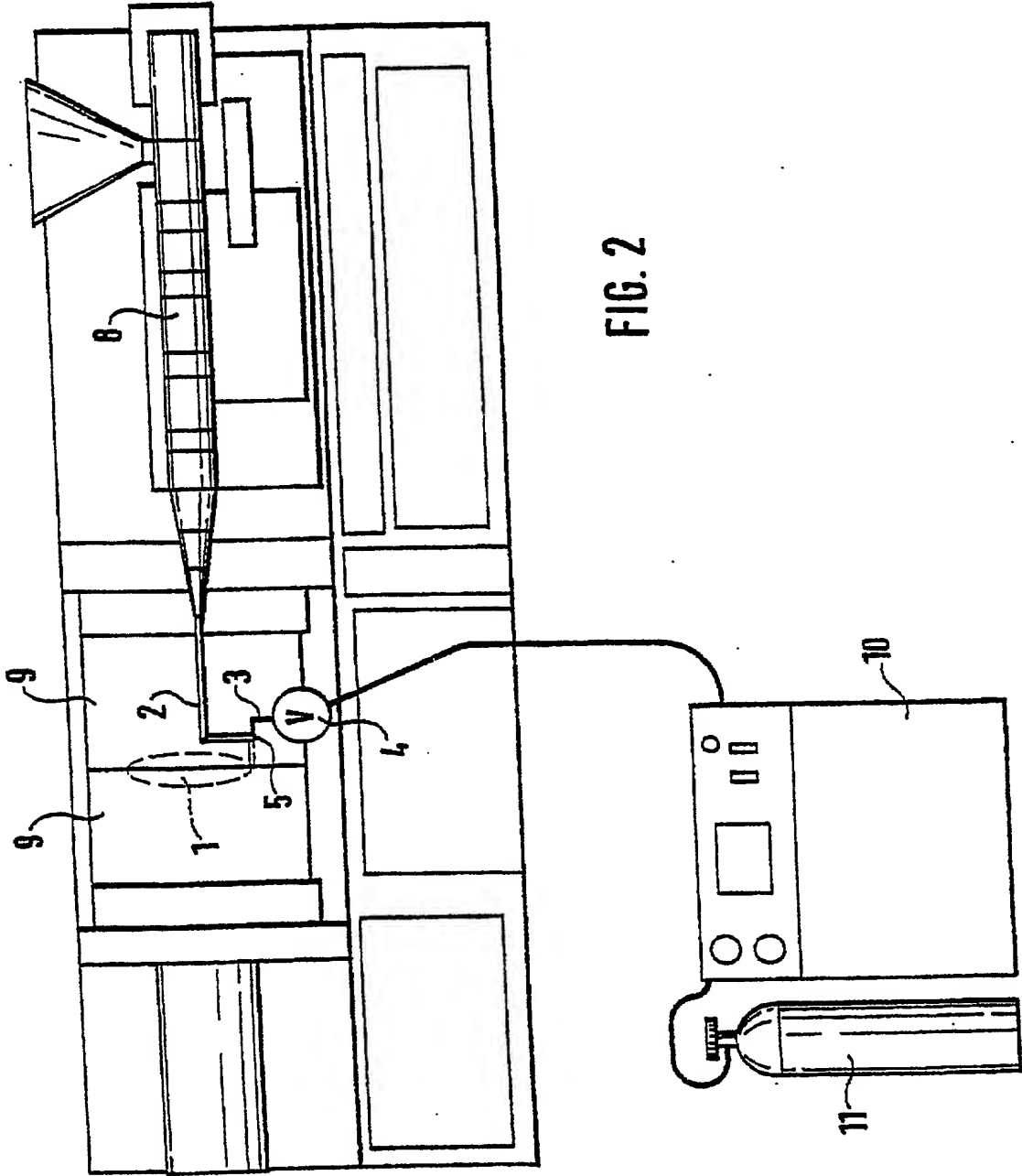


FIG. 2

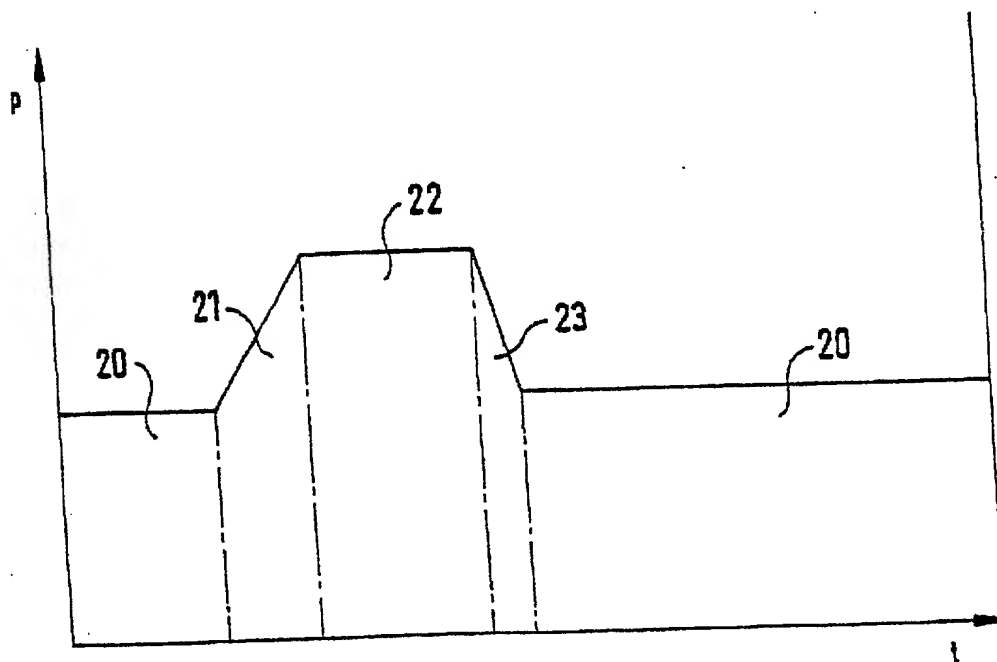


FIG. 3

FIG. 3

FIG. 4a

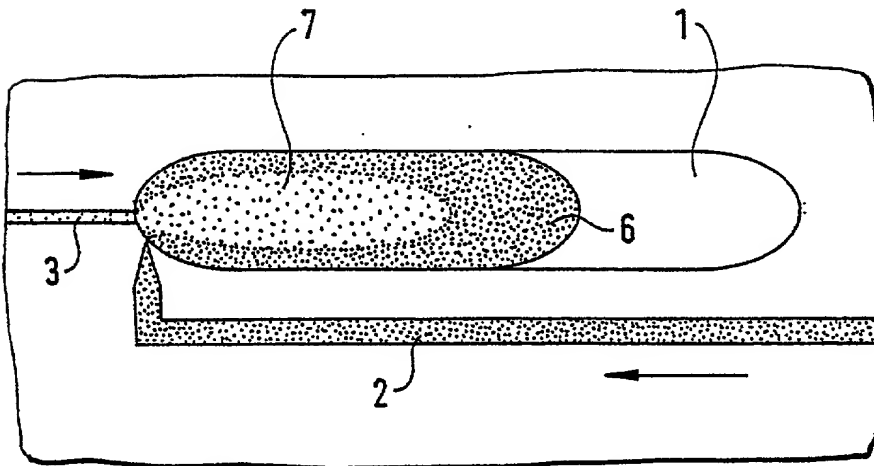
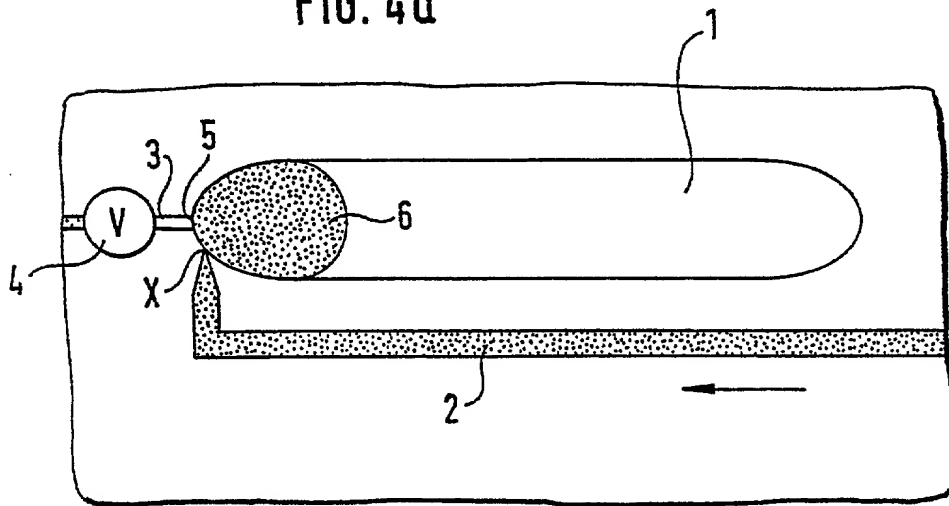


FIG. 4b

FIG. 4c

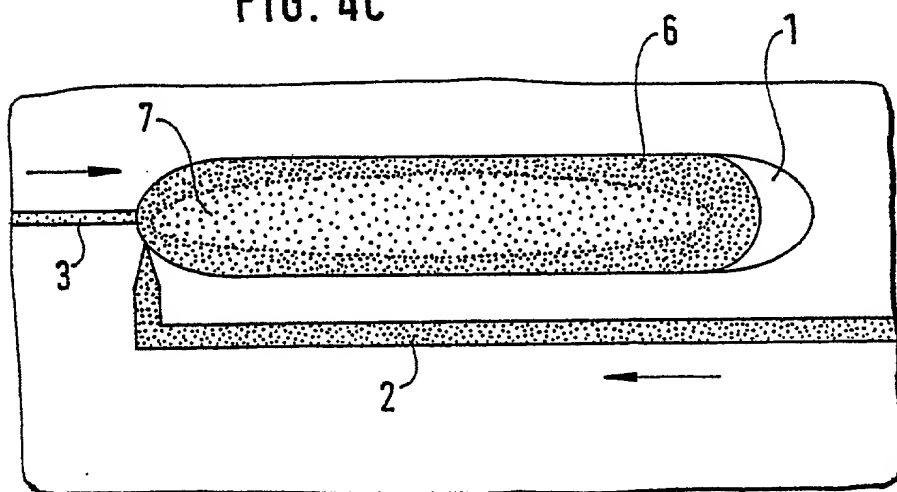


FIG. 4c

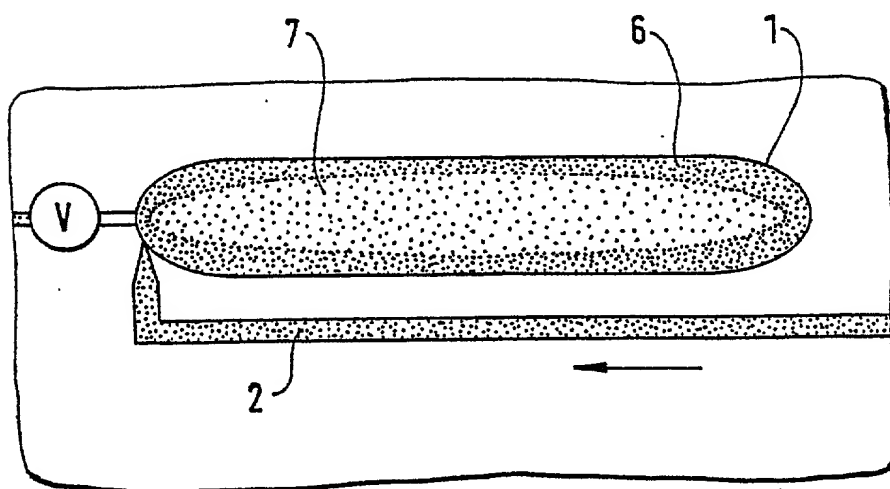


FIG. 4d

FIG. 4d

COMBINED DECLARATION AND POWER OF ATTORNEY

**(ORIGINAL, DESIGN, NATIONAL STAGE OF PCT, SUPPLEMENTAL, DIVISIONAL,
CONTINUATION, OR C-I-P)**

As a below named inventor, I hereby declare that:

TYPE OF DECLARATION

This declaration is for a national stage of PCT application.

INVENTORSHIP IDENTIFICATION

My residence, post office address and citizenship are as stated below, next to my name. I believe that I am the original, first and sole inventor of the subject matter that is claimed, and for which a patent is sought on the invention entitled:

TITLE OF INVENTION

METHOD FOR PRODUCING PHYSICALLY FOAMED INJECTION MOULDED PARTS

SPECIFICATION IDENTIFICATION

The specification was described and claimed in PCT International Application No. PCT/EP00/02258 filed on March 15, 2000.

ACKNOWLEDGMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information, which is material to patentability as defined in 37, Code of Federal Regulations, Section 1.56.

PRIORITY CLAIM (35 U.S.C. Section 119(a)-(d))

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

Such applications have been filed as follows.

**PRIOR PCT APPLICATION(S) FILED WITHIN 12 MONTHS
(6 MONTHS FOR DESIGN) PRIOR TO THIS APPLICATION
AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. SECTION 119(a)-(d)**

INDICATE IF PCT	APPLICATION NUMBER	DATE OF FILING DAY, MONTH, YEAR	PRIORITY CLAIMED UNDER 35 U.S.C. SECTION 119
PCT	PCT/EP00/02258	15 March 2000	yes

POWER OF ATTORNEY

I hereby appoint the following practitioner(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

APPOINTED PRACTITIONER(S)

REGISTRATION NUMBER(S)

Jodi-Ann McLane

36,215

Michele J. Young

43,299

Elliot A. Salter

17,486

I hereby appoint the practitioner(s) associated with the Customer Number provided below to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.

SEND CORRESPONDENCE TO

DIRECT TELEPHONE CALLS TO:

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401-421-3141

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321 South Main Street
Providence, RI 02903-7128
US

Customer Number 000987

05336756-03701
T0260-9529E660

DECLARATION

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

SIGNATURE(S)

100 Ulrich STIELER

Inventor's signature

Date 03.09.2001

Country of Citizenship Germany

DEX

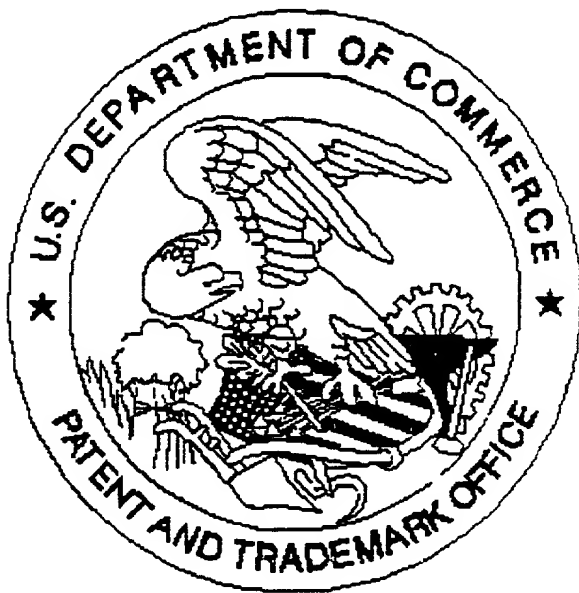
Residence Goslar Germany

Post Office Address Fontaneweg 1, Goslar D-38642 Germany

Ulrich Stielér

09936756-091701

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